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BUREAU OF AGRICULTURAL AND INDUSTRIAL CHEMISTRY



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of the
REGIONAL RESEARCH LABORATORIES
in connection with
the Seventh Annual Meetings
of the

AGRICULTURAL EXPERIMENT STATIONS RELATIONS COMMITTEES

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WESTERN REGIONAL RESEARCH LABORATORY

T. L. Swenson, Director

ALFALFA UTILIZATION INVESTIGATIONS

DEHYDRATION

Alfalfa Meal Standards

As a part of a general consideration of the status of the alfalfa dehydration industry, a summary of the history and current situation on grades or standards for alfalfa meal has been prepared. Grades for alfalfa hay have been issued by the USDA but no standards or grades for alfalfa meal have been established. A tentative standard was circulated in 1935 but it has not become official. The industry has formed its own designations of grades but most purchasers buy on the basis of green color. OPA ceiling prices are determined on protein content. It is judged that as the sellers' market loses its strength, uniformity of grade designations, of standards and of labeling will become imperative if alfalfa meal is to maintain its relative position in competition with other protein feeds. Some of the work being done at this Laboratory will be useful in establishing methods and procedures upon which grade standards can be established.

Alfalfa Dehydration Plants

In order to have complete information on the location and extent of the industry a list and map of alfalfa dehydration plants in the United States has been compiled.

EXTRACTION, STABILIZATION, ANALYSIS OF CHLOROPLAST PIGMENTS AND OTHER CONSTITUENTS

Stabilization of Carotene in Oil Solutions

A room-temperature experiment (25° C.) set up approximately a year ago to test antioxidants for their ability to protect carotene (provitamin A) in oil solutions has been completed. This work has as its objective the development of a better means for storing carotene which may be obtained from alfalfa. The room temperature storage test showed general agreement with the more rapid elevated-temperature tests (40° C. and 70° C.) although with the better anti-oxidants the carotene protection at the lower temperatures was somewhat better than that indicated by the rapid test.

Nordihydroguaiaretic acid, a material of vegetable origin and which has been accepted for use in lard by the War Food Administration was the best stabilizer tested. It held carotene losses in oil solution to only 20% for a year at 25° C. as compared to an equal loss in similar solutions within a few days without added antioxidants.

Analysis for Carotene Isomers

Work is under way on the improvement of methods for determining carotene isomers in fresh alfalfa. Various methods now in use are being compared and although an entirely satisfactory method has not been found, progress is being made. If a rapid accurate method of analysis is developed, it will be of value in determining the effects of handling alfalfa (field drying, artificial drying, storage, extraction) on its vitamin A activity.

FRUIT UTILIZATION INVESTIGATIONS

FREEZING PRESERVATION

Fruit Purees

It has been found possible to inactivate the enzymes responsible for darkening of apricot purees by passage through a plate type heat exchanger for rapid (15 sec.) heating to 189-206° F. The flavor is especially good when the puree to be heated contains 25% sugar. The sugar seems to prevent the development of a cooked flavor. Purees from Moorpack apricots obtained at Wenatchee, Washington, were judged to have a fine flavor.

Bing cherry puree was prepared from split cherries. The object of this work which is being done in cooperation with the Washington State Experiment Station is to develop a more successful process for utilizing cherries split by rain. Such damage frequently causes serious losses to cherry growers. The length of time cracked fruit can be left on the tree and the time required for it to spoil after being picked are being studied on frozen samples of fruit which were held for different periods of time in these two states. Puree also was prepared from shrivelled cherries, the so-called "raisins" and fruit from pollenizing trees. It was shown that an excellent Velva Fruit can be made from these cherry purees. They have the advantage that the relatively high natural sugar content of sweet cherries makes the addition of large amounts of sugar unnecessary.

Cantaloupe purees from a melon type grown extensively in the Imperial Valley were found to be equally as suitable for making Velva Fruit as the cantaloupes used previously. Cantaloupe purees are especially interesting because they are made from melons too ripe to ship. By making a puree and freezing it a large quantity of food material can be saved which otherwise would be wasted. This situation also places cantaloupe puree in a very favorable price range.

Other fruit purees which were prepared include rhubarb, strawberry, peaches, boysenberry, blackberry, raspberry, black and red currants, gooseberry, plums, cherries and nectarines. Various processing methods were employed depending upon the characteristics of the fruit and its susceptibility to oxidation. The general objective being to develop a satisfactory technique for different kinds of fruits so that this process can be used for salvaging surplus, and other fruit which is unmarketable because of size, shape or minor blemishes and is for that reason not acceptable as whole or canned fruit.

Velva Fruit

The experimental production of Velva Fruit has been continued to test the process on different fruits and blends. For example, apricot and sour cherry blends were found to be very good. Indications are that the industrial production of Velva Fruit will be

greatly increased as soon as sugar again becomes more freely available. Present commercial production is of the order of 50 to 100 thousand gallons per month as judged from the incomplete reports we have obtained.

Pie Fruits

The pack of frozen apricots in California alone has increased from 55,000 lbs. in 1940 to 34,800,000 lbs. in 1944 and that of peaches from 97,000 to 22,700,000 lbs. Most of these frozen fruits were used in pies. A large share of them were treated with a sodium bisulfite bath using a procedure developed at the Western Laboratory.

During the past year an improvement was made which resulted in greatly speeding up the penetration of sulfur dioxide into the fruit. This method consisted of adjusting the sodium bisulfite dipping bath to a pH of 3 by adding hydrochloric acid or 0.25% citric acid. The correct amount can be readily determined without instruments by noting the point at which the first traces of sulfur dioxide fumes can be detected.

Experimental packs were prepared which seem to be very promising. The process used is as follows:

1. A short (45 sec.) steam blanch to inactivate skin enzymes.
2. A sodium bisulfite solution dip.
3. A dip in a solution of 1% ascorbic acid and 2% salt.
4. Submerging in a sugar syrup containing ascorbic acid.

Less than half the sulfur dioxide (from sodium bisulfite dip) was required. The flavor was better and removal of sulfur dioxide easier. The steam blanch is so short that it does not cause a cooked flavor, soften the fruit or entail a significant leaching loss. Salt and ascorbic acid have a synergistic action in this application. The suggested dip will prevent darkening for from four to six hours after defrosting in either apricots or halved peaches. The process outlined above is an improvement over previous methods which processors agree are not entirely satisfactory.

New Pie Pack

A new and improved pie pack has been developed. It consists of coating otherwise untreated fruit slices with a puree-ascorbic acid mixture. The thickened puree containing the required sugar for the pie and ascorbic acid coats the fruit slices and effectively prevents them from darkening during frozen storage and subsequent defrosting.

Freezing Technique for Italian Prunes

A study has been made and a process developed for freezing this fruit for later use in preserves and pies. A variation of the process can also be used for preparing a retail consumer's pack. In general the process is similar to that for peaches and apricots although the nature of this fruit makes "slip peeling" desirable for the consumer's pack. Italian prunes are an important crop in

the Pacific Northwest and it is believed that improvements in freezing preservation will be of considerable value in marketing this fruit.

A New Antioxidant

Preliminary experiments indicate that dihydroxymaleic acid acts as an antioxidant and behaves similarly to ascorbic acid in preventing darkening of frozen fruits during defrosting. No evidence of its possible toxicity has been found in the literature. An advantage might be its lower cost.

Coliform Organisms in Orange Juice

The increasing industrial importance of frozen "single strength" orange juice has focused attention on its microbiological content. Fairly extensive bacteriological studies were made in five plants in Southern California. It was found that plate counts ran from 40,000 to over 1,000,000 per cubic centimeter of freshly frozen juice. There was a definite relationship between the quality of oranges being used, general sanitation and the bacterial content of the finished juice. It appears that organisms in many respects similar to Escherichia coli are often present in commercial orange juice but that they are not typical E coli. Since orange juice is not favorable to the growth of bacteria it seems probable that the organisms were located originally in the less acid peel and are carried into the juice during the mechanical extraction process. The use of clean sound fruit and good plant sanitation are of primary importance in this as well as in the production of all other frozen fruit and vegetable products.

Blanching and Cooling

Temperature measurements indicate that the insulating effect of the fruit tissue in large pieces such as peach halves is such that blanching can be accomplished by a shorter heating period with a subsequent wait for a short time to allow the interior to come to the required temperature. This will prevent some of the bad effects of overblanching the exterior. It was also determined that peach halves cooled almost as quickly in air as they did in vat cooling which is widely used in industry. At least one large processor is now installing cooling equipment of this new air cooling type. Vat cooling has the disadvantage of causing appreciable leaching losses. In connection with these studies, an investigation has been made regarding the extent of leaching losses during blanching and cooling. This has necessitated a study on and development of methods for determining the extent of these losses.

Experimental Centrifugal Deaerator

A centrifugal deaerator has been designed and is now in process of being made. Improved equipment for removing air from fruit and vegetable juices prior to freezing or canning would be very helpful in obtaining a product having improved flavor and vitamin retention.

DEHYDRATION

Spray drying

Peach extract, tomato paste, grapefruit juice and berry juice have been dried in the large spray drier in the pilot plant. Difficulties with sticky powders and mechanical troubles are being

gradually and somewhat laboriously eliminated. Modifications in the powder collecting system have been found to be necessary and will be made as soon as possible.

Drying Rates of Raspberries

Nomographs representing the drying rate characteristics of raspberries have been prepared. This basic information should be useful to processors in commercial operations.

PECTIN

Production of Low-Methoxyl Pectin by a New Method

Acid precipitation of alkali-hydrolyzed low-methoxyl pectin seems to be a very promising new method by which this important material can be produced at a cost appreciably less than by the currently used alcohol method. Plans for the production of low-methoxyl pectin from citrus peel using a counter current continuous process with this method have progressed to the point where a large commercial concern is erecting pilot plant facilities for research in cooperation with the Western Regional Research Laboratory. Better than 90% recovery can be obtained under conditions used in the laboratory work on this process.

Utilization of Apple Thinnings

Apple growers of the Pacific Northwest have expressed interest in the possibilities of economical utilization of the large tonnage of apple thinnings which are dropped on the ground or destroyed as a pest-control measure. Two boxes each of early and late thinnings of Delicious and Winesap Apples (8 boxes in all) were obtained from the Washington State Experiment Station at Wenatchee, Washington. Analysis and laboratory extraction of pectin indicates that these thinnings may be useful. An economic analysis of the problems is under way.

Chemical and Physical Studies on Pectin

Studies on such topics as gel strength, molecular weight, viscosity and acylation have been made. While this work is highly theoretical and quite fundamental in nature it nevertheless is essential to the solving of many very practical problems connected with the production and development of new products and uses for pectin.

Jellied Fruit Salad

Based on a formula developed at this Laboratory using low-methoxyl pectin the Quartermaster Department has procured 25 million cans of a jellied fruit salad for use by the Armed Forces.

Pectin Fibers and Films

A study of the film and fiber qualities of pectinic acids is under way. Fibers having tensile strengths in the range of 60,000 lbs. per sq. inch and films of 17,000 lbs. per sq. inch have been obtained. The fibers show excellent orientation as determined by X-ray methods and are fairly elastic. The films are flexible but have shown low shear resistance.

PROTEIN, AND VITAMIN-RICH FOOD OR FEED SUPPLEMENTS

Torula Yeast Investigations

Further successful pilot plant study has been conducted on the production of torula utilis yeast from pear waste. This work was done at Olympia, Washington, in cooperation with the Olympia Canning Company and the Oregon State Experiment Station. About 550 pounds of crude dry yeast was produced for use in feeding tests to be conducted in cooperation with Oregon State College Agricultural Experiment Station. One limitation in the commercial adoption of this process is the lack of satisfactory equipment for separating the juice for fermentation from such materials as pear waste and soft apples. To date there is no indication that anyone has developed a mechanical unit that can accomplish this job satisfactorily and economically.

ENZYMIC PREPARATIONS

Pectic Enzymes

The enzymes that catalyze the hydrolysis of pectic substances have been studied. New information regarding the properties of pectinesterase and polygalacturonase has been obtained. The enzymic hydrolysis of pectic substance has been shown to involve action of the esterase and galacturonase in sequence. The basis has been laid for more effective use of the pectin enzymes system in juice clarification. Citrus processing wastes appear to be a likely commercial source of pectinesterase.

Amylase and Maltase in Orange Peel

The presence of amylase in orange peel was confirmed and it was discovered that maltase also is present. A new enzyme, acetylsterase, also was discovered. Like much other basic information the significance of these findings is not known at the present time.

ISOLATION, ANALYSIS, AND CHARACTERIZATION OF CONSTITUENTS

X-Ray Analysis of Sodium Pectate

Work done on this subject has afforded the first opportunity for deducing reliable structural data on the polygalacturonide chain. The part played by the non-uronide material in determining the properties of pectic materials such as gel strength, acid behavior, etc., is of considerable interest to the chemist.

ASCORBIC ACID FROM WALNUT HULLS

Vacuum Concentration of Hull Extracts

Approximately 2,500 gallons of dilute hull extract were concentrated to 80 gallons for further laboratory investigation. Waste walnut hulls contain appreciable quantities of ascorbic acid and seem to be a logical source for obtaining this material provided that an economically feasible process can be developed.

POULTRY UTILIZATION INVESTIGATIONS

DEHYDRATED EGG STUDIES

Effect of Acidification

The control of acidity during the drying and storage of eggs has been demonstrated to extend by at least four times the useful "shelf life" of the product. While the beating properties of unacidified spray dried eggs are very poor those that had been properly acidified before drying are essentially equal to fresh eggs. The process consists of acidifying the broken out eggs with Hydrochloric Acid to a pH of approximately 5.5 before drying and mixing enough sodium bicarbonate with the dry powdered eggs to neutralize the acid. Upon reconstitution of the powder with water the reaction between the acid and soda forms a small amount of sodium chloride. The salt thus introduced is barely detectable. This method appears to make possible notable extensions in the usefulness of spray-dried eggs in the baking industry.

Components Responsible for Loss of Palatability

Evidence has been obtained that changes in the phospholipids during the storage of dried eggs contribute to deterioration in palatability. Considerable fundamental work has been done on the chemistry of these changes. It has been and will be useful in devising methods for preventing some of the undesirable changes which are not directly related to the moisture content of the stored powders.

Vapor Pressure of Egg Powders

Extensive studies have been made on this subject. The results provide essential data for the production of low-moisture egg powder of high keeping quality. They are also useful in the determination of the moisture content of dried eggs by means of a rapid vapor pressure method devised at this Laboratory. This method recently was adopted by the U. S. Quartermaster Corps for use in their testing and procurement work.

Color Changes During Storage

Color changes from yellow to brown which develop during storage of dehydrated eggs have been studied spectrophotometrically. These color changes have been correlated with loss in palatability and were shown to be accelerated at the higher storage temperatures. Evidence was obtained that the brown color is due to "aldehyde-amino" reaction products. Basic information has been obtained regarding the chemical nature and causes of changes in the constituents responsible for flavor deterioration of stored dehydrated eggs.

Antioxidants for Egg Oil

Of a series of amino acids tested as antioxidants for egg oil, proline proved to be the most effective. This search for antioxidants may lead to the discovery of additives suitable for prolonging the storage life of dried eggs.

Salt Soluble Fluorescence Test

It has been shown that the widely used salt-soluble fluorescence test of quality is not reliable as an objective substitute for

palatability tests for stored egg powders. Evidence has also been obtained that the products of bacterial metabolism which are extractable by salt solutions may contribute to the fluorescence of egg powders.

Raw Materials

The quality of the eggs used for drying has been shown to influence the keeping properties of the product.

Moisture Content

Decrease in moisture content from 6.0 to 0.5% resulted in increases in the shelf life of the powders. No decrease in palatability took place in egg powders dried from the frozen state when stored for 8 months at 4° C. The time during which similar egg powders, stored at 20° C., remained acceptable for scrambling was approximately 8 months compared to 1 month when stored at 36.5° C. Considerable data has been accumulated to show the importance of low moisture content and low temperatures in avoiding deterioration of dehydrated eggs.

Free Fatty Acid Analysis

The A.O.A.C. method for the acidity of the ether extract of eggs has generally been used for measuring changes in the acidity of dried egg powders during storage. It was found that the method is inadequate for measuring changes in true fatty acidity of egg powders. A procedure for eliminating the errors in the A.O.A.C. method has been developed.

FREEZING PRESERVATION

Resazurin Test

This test has been shown to be useful for readily detecting high bacterial count in frozen eggs. As little as 0.1% of bad egg could, when mixed with good egg, be detected. The speed, simplicity and cheapness of this test should make it very useful in the egg breaking industry as a quality control method. There is a wide field for its application.

LYSOZYME FROM EGG WHITE

New Isolation Method

A new and improved method for isolating lysozyme consists of adjusting the pH and salt concentration, storing at near freezing temperature and separating the crystalline lysozyme. Yields of 85 to 90% are obtained. This method makes it possible to readily and economically obtain this interesting antibacterial agent for clinical testing by cooperating agencies.

Crystallographic Properties

The crystallographic properties of lysozyme have been determined. Since very few proteins crystallize and since they cannot be characterized by melting points it is important to know the crystallographic and optical properties for identification purposes and for comparison with other proteins.

FIBERS, PLASTICS, AND ADHESIVES FROM EGG AND FEATHER (KERATIN) PROTEINS

Physical Properties of Dispersed Feather Keratin

Progress has been made in studies on the molecular size and configurations of dispersed chicken feather keratin. This keratin dispersed in a neutral aqueous solution of an alkylbenzene sulfonate with sodium bisulfite forms a stable complex. It has been examined for the following: ultracentrifugal sedimentation, osmotic behavior, diffusion viscosity and specific refractive increment. The average molecular weight of the keratin portion of the complex has been determined to be of the order of 34,000 to 40,000 thus approximating egg albumin in size.

Fiber Stretching and Curing

Improvements in the mechanics of continuous stretching have been made and better conditions for curing the stretched fiber developed. These advances have resulted in an increase of from 3 to 4 times in the wet strength, improved elasticity and better boil resistance.

ISOLATION, ANALYSIS, AND CHARACTERIZATION OF CONSTITUENTS

Effect of Dry Heat on Proteins

It has been found that dry heat in the range 100 to 200° C. on egg white, feathers and other keratins causes decreases in solubility, digestibility by enzymes, amino nitrogen, total basic groups, and equilibrium water content at 70% relative humidity. The most marked changes begin to occur at 150° C. A study of the effect of dry heat on protein was started when it was noted that keratins after heating to 415° F. could be mixed with phenolic resins to produce plastics having improved properties. The results are also being applied to fiber treatment.

Surface Area of Egg Albumin

Measurements of the surface area of egg albumin accessible to water vapor when calculated in terms of the recently developed Harkins-Jura adsorption theory indicates an area of approximately 190 meters per gram of egg albumin. This agrees quite well with a surface area of 210 meters as calculated by the Frumauer-Emmett-Teller theory. The surface area is a useful constant for characterizing water adsorbing materials.

VEGETABLE UTILIZATION INVESTIGATIONS

DEHYDRATION

Storage Studies

In cooperation with the Chicago Subsistence Laboratory of the Quartermaster Corps much was accomplished towards the commercial application of in-package (lime-package) desiccation of dehydrated vegetables. A package for the lime which proved practical is an ordinary spiral-wound fiber carton having seamed-on metal ends. Specifications for a suitable lime for this purpose were determined. Although the war ended and the lime-package did not go into large scale

commercial use, its development has been completed to a stage where it can be very helpful in maintaining the quality of dehydrated vegetables stored in tight containers.

It has been demonstrated that easily controllable factors such as moisture content, and composition of the package atmosphere are effective in the prevention of rancidity and staleness in many kinds of dehydrated vegetables.

Comprehensive storage studies on various dehydrated vegetables have been continued. Most of the remaining edible samples are in the room temperature series (some after about 18 months). At the higher temperature storage (90° and 100° F.) there remains in good condition only the samples of very low moisture content (attained by in-package desiccation).

Leaching Losses

The loss of soluble solids during blanching prior to dehydration or freezing is a serious problem. Considerable work has been devoted to establishing accurate methods for determining the extent these losses and in devising equipment and procedures which will reduce them to a minimum.

Standard Samples for Ascorbic Acid Tests

In collaboration with the University of California, 700 lbs. of spinach was dried to a moisture content of 2.5% and canned in an atmosphere of carbon dioxide. The product is to be used as an ascorbic acid reference standard by State Experiment Stations.

Estimation of Water-Soluble "Brown" Color

A color standard for measuring the "brown" color in dehydrated white potatoes has been found. The standard is a glass light filter Corning No. 4303. In the spectral region of interest the light transmission curve of this filter is similar in shape and slope to that found for extracts of dehydrated white potato at the pertinent stages of deterioration. This work was done at the suggestion of the Quartermaster Corps' Coordinator of white potato dehydration studies. The use of this standard will be very helpful in aligning results of various workers using different colorimeters. The "brown" color content in dehydrated white potatoes can be used to determine the extent of heat damage during the drying process.

Density and Mushiness in White Potatoes

A positive correlation has been shown between density of the raw potato and a tendency to mushiness upon cooking. This fact may be useful in selecting suitable potatoes for dehydration.

Spray Drying

The relationship of equilibrium, vapor pressure and "Sticky point" temperature was investigated for 15 different spray dried fruit and vegetable powders. "Sticky point" refers to the lowest temperature at which powder particles tend to adhere to each other and lose their free-flowing characteristics. It is definitely related to the tendency of the powder to stick to various parts of the spray drier.

For spraydried green pea and soybean powders the indicated sticky points were above 140° C. at 3.7 and 3.3% moisture, respectively. For spray-dried tomato powder of 3% moisture this point is 62° C. Tomato powder containing 1% (dry basis) added "methocel" showed a sticky point about 20° C. higher than that of untreated material at the same moisture content. It also was found that the addition of small quantities of rind solids to citrus juices materially raises the sticky points of the resulting powders.

Measurements of equilibrium, vapor pressure and sticky point temperatures are indicative of the ease with which a particular fruit or vegetable can be spray dried. Indications are that green pea and soybean powder can be obtained without difficulty while berry and soft fruit powders will be difficult. Tomato powder appears to be with a borderline group. The employment of additives appears promising as a means of elevating the sticky point. Much work is planned for the coming year on spray drying problems.

Rotary Peeler

A laboratory-size rotary peeler suitable for peeling peaches, potatoes and other root crops has been designed and procured for use in experimental work on improvement in chemical peeling methods. If preliminary indications are borne out this peeler may be of importance to industrial processors as an improvement over present equipment.

Thermal Properties of Dehydrated Vegetables

The specific heats and thermal conductivities of dehydrated vegetables and egg powders were measured. These data are necessary for heat transfer calculations relating to the design of dehydration equipment and to the temperature history of dehydrated products subsequent to packaging.

Dielectric Properties of Dehydrated Carrots

Measurements were made to establish a basis for the determination of moisture content by electrical methods. From these measurements it was established that the power factor is the most sensitive of all the variables, to changes in moisture content and is the most suitable to be measured and related to the amount of moisture present. These data provide information essential for the design of an electrical device suitable for the rapid and accurate measurement of the moisture content of dehydrated foods. The need for such an instrument has existed in the industry for a long time.

Vitamin A Analysis

Cooperative work with the Pharmacological Laboratory has shown that the chemical and bioassay methods on dehydrated vegetables are in excellent agreement if the carotene is extracted and fed to the rats as an oil solution in the bioassay test. However, the chemical and biological method may show a marked discrepancy in the vitamin A value of dehydrated carrots. This indicates that the carotene is not all biologically available to the rat. In dehydrated carrots stored at 98° F. and 120° F. the bioassay method gave only one-third of the chemically determined value. The nutritional aspect of these findings are obviously important.

FREEZING PRESERVATION

Frozen Corn

During the 1945 season, samples of sweet corn were prepared at Prosser, Washington, in cooperation with the Washington State Experiment Station. Both Golden Bantam and Golden Cross Bantam at two stages of maturity gave a better product when blanched before cutting from the cob than when blanching was done after removal of the corn from the cob. Blanching of sweet corn on the cob appears to be a desirable commercial practice.

Frozen Peas

A large number of samples were packed this season at Farmington, Utah, in cooperation with the Utah State Experiment Station. Laboratory examinations on this year's pack have not been completed.

Evaluation of the 1944 pack, however, was completed. Of the three factors considered, maturity has the greatest effect upon the quality of the finished product because of its pronounced effect upon texture and flavor. A high degree of correlation was found between starch and total solids content and texture of frozen peas, while there is a poor correlation for the relationship between specific gravity of processed peas and these quality factors. On the basis of these and other considerations it is tentatively recommended that the starch content as determined by the Nielsen method be used as the basis for estimation of texture or maturity in grading work. The industry and the Production and Marketing Administration are interested in development of improved objective tests for quality in frozen peas.

Off Flavors in Frozen Peas

The development of these undesirable flavors appears to be one of the principal problems of the pea freezing industry. Based on the hypothesis that objectionable flavors in peas may be due to aldehydes or may parallel aldehyde content two separate techniques have been investigated for aldehyde determinations. These methods show promise of being very useful in future studies on the problem of "Off Flavors" in frozen peas.

Frozen Tomato Juice

Samples were packed in three kinds of containers, waxed tubs, plain tin cans and enameled tin. The juice packed in plain cans averaged 7% higher in ascorbic acid than those packed in waxed tubs and 4% higher than those in enameled cans. The frozen juice in waxed tubs, however, showed a significantly higher ascorbic acid content than the cooked product.

Coliform Bacteria in Frozen Vegetables

A large number of commercial samples of various vegetables were bought from different sources in the open market and analyzed for the presence of coliform bacteria. Results show that while bacteria giving presumptive tests, i. e., formation of lactose with acid and gas, are fairly numerous, completed tests prove that the incidence of E. Coli is quite low. While no judgement is offered as to tolerable limits of these bacteria it was shown to be important for any public.

health or regulatory authority to differentiate between "fecal" coli and the "grass" or Aerobacter organisms which will give a presumptive test but are so widely distributed as to be of comparative little sanitary import.

JUICE CONCENTRATES FROM VEGETABLE WASTES FOR USE IN THE PRODUCTION OF ANTIBIOTICS AND ENZYME PREPARATIONS

Subtilin

Progress has been made in the development of a method of isolating subtilin from B. Subtilis culture liquors which consist of asparagus butt juice as a base. Evaluation of subtilin is under way by the University of California through a cooperative agreement. Preliminary indications are that it may be useful in the treatment of two diseases for which there is at present no fully adequate method of treatment; namely tuberculosis and amoebic dysentery. Preliminary studies indicate also probable usefulness of the antibiotic for the control of some plant diseases. Other crude antibiotic preparations also are being investigated for this purpose. In this use it is not necessary to purify or highly concentrate the products, hence production costs will be much lower than for a material planned for medicinal use.

Gramicidin

Gramicidin, the more active component of tyrothricin, has been reacted with formaldehyde to yield a product which is less toxic and which apparently retains its desirable antibiotic activity. This is expected to extend the possible medicinal usefulness of the product.

Asparagus-butt juice has been shown to be useful as a culture medium for several antibiotic-producing organisms. The use of juice concentrates, made from waste butts, may be encouraged by the results of these researches.

ISOLATION, ANALYSIS, AND CHARACTERIZATION OF CONSTITUENTS

Spectrographic Analysis

A spectrographic method for the determination of calcium in asparagus juice has been developed. The method is being expanded for simultaneous determination of magnesium, manganese, copper and iron in any similar organic solution.

A spectrographic method has been applied to the simultaneous determination of potassium, magnesium, calcium, sodium, phosphorous and boron in dehydrated potatoes. The determinations were needed in connection with an investigation of factors causing variations in properties of potatoes on reconstitution and cooking. The procedure will also have many other applications in research on agricultural products.

WHEAT UTILIZATION INVESTIGATIONS

INDUSTRIAL NON-FOOD USES OF GLUTEN

A Gluten-Glycerol Rubber Like Product

Gluten plasticized sheets of uniform texture and pleasing appearance have been prepared from a gluten-glycerol mixture. The material has good oil, grease and hydrocarbon solvent resistance

although it is not resistant to water. It's possible use in printer's rolls has been suggested and is under consideration by one manufacturer of these rolls.

GLUTAMIC ACID

A new and improved method for the determination of glutamic acid was developed. This procedure is of considerable usefulness and interest to manufacturers and research workers using or producing glutamic acid.

GLUTEN SULFATE

Improved Production Process

Industrial interest has continued in this gel-forming reaction product from wheat gluten and sulfuric acid. A larger scale and improved process has been developed for the preparation of samples for evaluation in commercial applications in the food, pharmaceutical and other industries.

Chemical Studies

The sulfated derivatives of most proteins, unlike that of gluten, are soluble. They represent an entirely new field of material with possible uses as detergents, emulsifying agents, etc. Only preliminary work has been done but an interesting and potentially fruitful field of research has been uncovered.

ERGOT ALKALOIDS FROM FUNGI GROWN ON WHEAT

Work on this project has been discontinued and the results summarized for publication. Useful information has been obtained with regard to the nutritional requirements of the organisms for growth.

ISOLATION, ANALYSIS, AND CHARACTERIZATION OF CONSTITUENTS

Gluten Protein Composition

It has been determined that glutenin is a lipoprotein. When separated from gliadin without the aid of alcohol, glutenin contained approximately 20% alcohol-soluble lipids. Proof has now been adduced that the protein does not exist as such in wheat flour, but, instead, is formed during the manipulations required to form gluten. As flour is kneaded into dough, the lipids are brought into contact with the glutenin component to form difficultly-dissociable complexes.

An understanding of gluten protein composition is of distinct advantage in evaluating various methods of preparing gluten and for finding end uses. In addition, the knowledge that gluten is formed during the kneading of bread may be of importance to workers on problems of the baking industry.

WESTERN HEMLOCK BARK UTILIZATION INVESTIGATIONS

An extraction process has been developed using sodium sulfite solutions at 175° F. Larger amounts of tannin can be obtained in this manner, than can be extracted using water alone.

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NORTHERN REGIONAL RESEARCH LABORATORY

G. E. Hilbert, Director

AGRICULTURAL RESIDUES UTILIZATION INVESTIGATIONS

SUNDRY NEW PRODUCTS

Noreseal--A Cork Substitute

Pilot plant investigation on Noreseal, which is produced from peanut shell pith, glue, sugar, and paraformaldehyde, has been completed. The initial pilot plant operation was for the production of Noreseal in sheet form. A second pilot plant operation was developed to produce Noreseal by a dispensing method. The latter method has produced not only the best crown seal for bottle crowns used in the beverage industry, but because of its automatic operation the method effects a reduction in labor, operating, and capital costs. Practical bottling tests in a hot room, using crowns produced in pilot plant operation by the dispensing method, showed that Noreseal-filled crowns were superior to cork disc-filled crowns in that no leakers developed with Noreseal, while a rather high percentage of leakers developed with the cork controls. At the present time 70,000 crowns for field tests have been produced and distributed to several of the largest brewing companies, through the U. S. Brewers' Association. One crown manufacturing company in Indiana has built a manufacturing unit to produce Noreseal by the dispensing method, with a capacity of 250,000 crowns per 8-hour day. A great deal of interest has developed outside the United States in the possibility of manufacturing Noreseal.

Soft-Grit Blasting

In cooperative work by this Laboratory with the U. S. Navy, corncobs ground to pass a 10-mesh and retained on a 32-mesh screen, were found to be the most suitable soft-grit blasting material for cleaning carbon and oil deposits from airplane engines and parts. A mixture of 60 percent corncobs and 40 percent rice hulls was found to be even more efficient. By use of the soft-grit blasting method all extraneous material, excepting enamel and extremely hard scale, can be removed from machine parts without causing dimensional changes, cutting or pitting. Glass manufacturing companies are employing the method for removing scale from steel molds; a tractor company is utilizing it to clean aluminum foundry core boxes and molds; and one of the largest automobile manufacturers has shown considerable interest in the method for certain operations in reconditioning motors, and in making line-production operations possible to a greater extent in this branch of the business. About 500 tons of corncobs and 350 tons of rice hulls were used last year for soft-grit blasting purposes. A very large tonnage of corncobs and rice hulls should eventually be used annually for this purpose.

ALCOHOL AND OTHER LIQUID FUELS

Fermentation Studies on Xylose

Fermentation studies on the xylose obtained from the hydrolysis of corncobs have continued. It was found that technical xylose, when combined in equal portions with glucose to give a total sugar concentration of 6 percent, could be satisfactorily fermented by butyl alcohol bacteria to butanol, acetone, and ethanol. Crude xylose sirups could be fermented in the same manner if the xylose was previously adjusted to pH 8.0 and filtered. It was also found that crude xylose and xylose sirups obtained from the acid hydrolysis of agricultural residues may be fermented to butanol, acetone, and ethanol in the absence of other sugars, provided inhibitory substances are removed from the xylose or their formation during sterilization is avoided.

PLASTICS, ELASTOMERS, AND RESINS

Noreplast-R (R= Residues)

Laboratory and pilot plant work on development of thermosetting resins, containing 25 percent of phenol-formaldehyde and 50 percent agricultural residues ground dry to a flour passing an 80-mesh screen, was completed. Satisfactory molding powders have been produced in two ways. One is by the use of liquid phenolics precipitated, washed with water, and compounded with 22 percent of Vinsol or modified Vinsol soap as a plasticizer, and 50 percent agricultural residue flour. The flow and molding properties of molding powders produced in this way make them acceptable for industrial application under present molding conditions. The second method is based on the discovery that commercial dry phenolic resins can be adapted for production of Noreplast-R compounds by the replacement of the Vinsol-soap plasticizer with an equal amount of inorganic extenders, such as chalk, iron oxide, titanium oxide, diatomaceous earth, and the like. This latter type of molding composition will lead to a reduction in cost of about 2-1/2 cents a pound of molding powder, which is substantial. A variety of resins may be used as filler, but ground corncobs and ground rice hulls appear to give the best results.

Noreplast-L (L= Lignin)

A comprehensive program on the utilization of saccharification lignin in resinous binders and adhesives has been conducted. Lignin was treated with 21 different materials, including asphalts and extracts from petroleum refining. An ammoniated lignin appears to have the most promise of the mixtures investigated. If ammoniated lignin is compounded with 25 percent of a phenolic resin and inorganic extender (CaCO_3), a material is produced having strength characteristics comparable to the Noreplast corncob compounds of similar resin and extender content.

SYNTHETIC ORGANIC CHEMICALS AND INTERMEDIATES

Hydrogenation and Dehydrogenation Products from Furfural

In the production of dihydropyran, when tetrahydrofurfuryl alcohol is passed over an aluminum catalyst, satisfactory yields of about 60 percent, as reported in the literature, can be obtained only at low feed rates since higher feed rates caused carbonization and poisoning of the catalyst. We have discovered that the catalyst may be conditioned so that it is capable of producing conversions in the neighborhood of 90 percent at high feed rates. Since this method is believed to be new, patent applications have been made.

Following up a novel suggestion that it might be possible to hydrogenate methylfuran in the presence of a catalyst and an acid to produce 1,4-pentanediol, it has been discovered that this reaction can be made to proceed in 61.5 percent yields. Effective chemicals such as ethylene glycol diformate, formic acid, or acetic acid, to supply the hydrogen ions necessary for the splitting of the ring were also investigated. 1,4-Pentanediol is interesting as an intermediate in the manufacture of the new German polyurethane plastics. Further, it was found that by stopping the hydrogenation reaction when half the hydrogen is used up, a mixture of acetopropyl alcohol and 1,4-pentanediol is obtained together with some tetrahydromethylfuran. Acetopropyl alcohol is a valuable intermediate for the synthesis of the thiamin (Vitamin B₁) molecule. This method would appear more direct than some of the methods now used for producing acetopropyl alcohol. A patent application covering this development was prepared.

Derivatives Arising from Opening of Furan and Pyran Rings

Hydrolysis of dihydropyran in the presence of a small amount of acid results in high yields of hemiacetal of hydroxyvaleraldehyde. This chemical probably presents an opportunity for use as an intermediate not only producing other chemical compounds but also shows evidence of ability to polymerize to form viscous and resinous-like products. Hydrogenation of the hemiacetal under pressure over a nickel catalyst leads to the production of 1,5-pentadiol of high purity in almost quantitative yields, thus offering a new method for the production of 1,5-pentadiol.

A study of malarial intermediates is being made. Starting with tetrahydromethylfuran, a process has been worked out for producing Noval alcohol. Noval alcohol is indicated as a possible intermediate for the manufacture of Plasmochin and Atabrin, two of the important antimalarial drugs.

STRUCTURE AND OTHER BASIC INVESTIGATIONS

Saccharification Lignin

Investigations have been under way to obtain information on the properties of lignin obtained from the saccharification of corncobs by the Synthetic Liquid Fuels Process. This lignin, like the Scholler lignin from wood, seems to be quite unreactive. The actual lignin fraction of the crude material is soluble in 1 percent sodium hydroxide, but the fact that it cannot be precipitated from alkaline solution by carbon dioxide appears to indicate the presence of a carboxyl group which has not been observed in Scholler lignin. Furthermore, the crude lignin appears to contain about 20 percent of material which is neither alkali-soluble lignin nor degenerated cellulose. Solvent extraction of the crude lignin with ether has yielded a waxy, oily product which accounts for 3 percent of the 20 percent of unknown material. A study is being made of the composition of this fraction. Work is being conducted on the chlorination of the lignin in an attempt to produce substituted chloro compounds. If this can be done the product might have some value as an insecticide or fungicide.

CELLULOSE PULPS FOR PAPER AND BOARD MANUFACTURE

General

The work done by the Laboratory has created a great deal of interest in the fine paper industry. The possibility of utilizing agricultural residues for pulp manufacture is becoming a concern of this group, partly because of the increasing costs of obtaining wood pulp and partly because industry is looking for new and improved products.

9-Point Corrugating Strawboard

Laboratory and pilot plant work on a process for producing an improved 9-point corrugating strawboard has proceeded so successfully that a large corporation has stated that this process offers possibilities of being the outstanding improvement in strawboard over a period of many years. Straw is chopped to a definite size and then given a mild alkaline digestion. The under-cooked straw is defibered in a double-disc, attrition mill-type refiner, resulting in the production of thin fiber bundles similar in appearance to the coniferous pulp fibers. These straw fiber bundles when mixed in suitable proportions with regular lime-cooked straw pulp produce a strawboard with highly improved tearing resistance and other properties. Arrangements have been made whereby the Laboratory is cooperating in carrying on a mill run test early in 1946.

BUILDING AND CONSTRUCTION MATERIALS

Insulation and Hardboard Fundamental Fiber Studies

In spite of the fact that insulating boards have been manufactured for more than 25 years, there is practically no information in the literature concerning fundamental fiber properties, and techniques for producing insulating building products. The same is true of hardboard panel manufacture. It is possible to produce from agricultural residues rather stiff, long fibers of a character not possible to produce from wood. In order to carry out an investigational program of fundamental fiber studies, several hundred pounds of straw fiber was cooked and fractionated into fiber lengths of approximately 1", 3/4", 1/4", and less than 1/4" in size. It was found that in preparing the boards, the use of a standard consistency of 99.5 percent water and 0.5 percent fiber made it possible to duplicate results accurately. The experimental program continues.

CORN, WHEAT, AND OTHER CEREAL CROPS UTILIZATION INVESTIGATIONS

COMPOSITION AS AFFECTED BY VARIETY, BREEDING ENVIRONMENT, AND MATURITY

Chemical Composition of Hybrid Corn

Analytical studies at this Laboratory have shown that inbred lines used in developing commercial hybrid corn seed govern the chemical composition of corn as such inbred lines govern yield and character of growth. This allows the corn breeder to produce hybrid corn which has the desirable properties of high oil or high protein in addition to the desirable agronomic qualities of high yield, resistance to disease, uniformity, and the like.

SUNDRY NEW PRODUCTS

Starch Sponge

The starch sponge prepared at this Laboratory has elicited interest from two sources, viz., research workers in medical schools and pharmaceutical companies who wish to evaluate it as a surgical dressing, and by food processors, confectioners, starch distributors and miscellaneous manufacturers. As an internal surgical dressing it is absorbable by the body and can be used as an external surgical dressing for burns. For food uses, it has been shown to be superior as an adjunct to add crunchiness to candies and as a tasteless carbohydrate source in Army rations or other prepared foods. The addition of 20 percent soybean flour or 20 to 50 percent wheat flour enhances its flavor.

Microscopic studies of the sponge revealed whole globules, globules which had undergone vacuolation, and myelin forms all of which are typical of coacervated colloid systems. This evident coacervate nature of the sponge provided a clue which led to further work on starch coacervates, and in turn resulted in a tentative theory of the formation of starch granules in the plant by coacervation of the carbohydrates with protein and fatty materials.

ALCOHOL AND OTHER LIQUID FUELS

Saccharification

In the experimental program on the reduction of saccharification costs, fermentations were carried out with (a) malt under various conditions of cooking, (b) additives to improve the efficiency of malt, (c) fungal amylases, and (d) acid as the saccharifying agent.

(a) Influence of cooking: By the application of a double cooking procedure great improvement could be obtained in saccharification. Mashers prepared by this technique fermented to completion in approximately 40 hours and gave slightly higher yields of alcohol than did mashers prepared by the conventional malting practice.

(b) Malt adjuncts: Papain has been shown to stimulate alcoholic fermentation, and its incorporation in grain mashers to result in a decrease in the malt requirement. A search conducted for other proteolytic enzymes of this type and a survey of more than 200 bacteria and fungi reveals that many of them show a high proteolytic activity. When added as a supplement to fermentations, incorporating a sub-optimal level of malt, several of these cultures showed stimulation equivalent to that obtained with papain.

In a study made of the effects of adding individual as well as complete mixtures of amino acids to alcoholic fermentations, it was found that no single amino acid gave more stimulation than might be expected from the added amino nitrogen. Mixtures of amino acids such as those contained in casein hydrolysates, however, gave excellent stimulation comparable to that imparted by papain.

(c) Fungal amylases: In an attempt to find sources of amylolytic enzymes which can completely replace malt, a survey has been made of amylases produced by 250 fungi cultured in thin stillage. Several strains have been found which form significant quantities and these enzymes are being employed in fermentation studies. One culture especially elaborates unusual quantities of amylase. Culture filtrates from this organism can totally replace malt in the alcohol fermentation of corn when used in a method developed at this Laboratory. Fermentation of mashers prepared in this manner has led to yields as high as 5.40 proof gallons of alcohol per bushel of grain. The results are significant in that fungal enzyme preparations capable of replacing malt may greatly reduce the cost of producing alcohol from grain.

(d) Alcohol from acid-saccharified grains: Conditions have been established for the saccharification by acid of corn and wheat and their subsequent fermentation to alcohol. Commercial preparations of mold bran and Diatane increase the rate of fermentation and the yield of alcohol. In fermentations using wheat, yields obtained were somewhat lower than those obtained when using malt for saccharification. Rye also has been fermented successfully after acid hydrolysis.

Physical Studies

Studies of the stability of alcohol-gasoline blends for various hydrocarbon-water mixtures have shown that, providing precautions are taken, blends containing 10 percent, or more, of ethanol are stable.

The solubility of oxygen and nitrogen in ethanol and other possible fuels has been determined, prompted by the fact that dissolved gases may be the cause of vapor-lock in aviation fuel systems, and this probably in systems used on the ground.

Vapor pressures and densities of ethanol-isooctane mixtures have been measured as a necessary step in the Laboratory's comprehensive determination of liquid-vapor relationships of ethanol-hydrocarbon systems.

Alcohol-Water Injection

Investigation of injection of alcohol and alcohol-water mixtures into the intake manifold of automobile engines has not been completed, but studies show engine performance can be improved by this method. A relatively high compression ratio, lean carburetor adjustment, and a fuel of lower octane rating than could ordinarily be used successfully under these conditions seem to give best results. Road tests and further laboratory work will be necessary to draw up recommendations.

Anti-Knock Agents

In studies leading to a comprehensive summary of octane numbers of ethanol motor fuel blends, it has been found that even though tetraethyl lead will lower the octane rating of pure ethanol, this is not the case in gasoline blends containing up to 25 percent of ethanol and moderate amounts of lead. Tests show that the addition of iron pentacarbonyl will raise the octane number of ethanol above 100, and a similar effect was noted when used in gasoline. It was found that the photosensitive iron pentacarbonyl could be stabilized. However, because of iron oxide deposits, its use as an anti-knock material cannot be recommended.

Corrosion

It has been shown that a large number of metals and alloys, such as might come in contact with motor fuels, when exposed to a 10 percent ethyl alcohol-commercial gasoline blend, do not corrode to any serious extent, nor to an extent greater than when exposed under similar conditions to commercial gasoline.

FIBERS, FILMS, PROTECTIVE COATINGS, AND ADHESIVES

Fibers from Zein

It has been found at this Laboratory that zein can be dispersed in aqueous sodium hydroxide solutions in the pH range of 11.5 to 12.5. Consequently, studies were undertaken to determine if textile fibers could be spun from these aqueous dispersions. The results showed that the zein fibers could be made by techniques and equipment similar in many respects to those used for spinning other protein fibers. The most important difference in the technique used for spinning zein fibers and that used heretofore on other fibers is in the formaldehyde pre-cure. The pre-cure is a controlled formaldehyde treatment carried out between the coagulating bath and stretching treatment and is the key to the comparatively high tensile strength of zein fiber. Small scale laboratory experiments showed that fibers could be produced which had the same dry tensile strength (1.2 gm. per denier) as wool and approximately 3/5 the wet strength (0.78 gm. per denier) of wool. By proper curing of the fiber with formaldehyde and acetic anhydride a moderate stretch could be retained along with relatively high wet strength and resistance to shrinkage. The process has been developed to operate on a continuous basis.

Plywood Adhesives

The great demand for waterproof plywood led to an increased use of phenolic resins for plywood adhesives previous to and during 1945. The Laboratory discovered that corn gluten meal (53 percent protein) or soybean meal (water solubles removed) could be used with phenolic resins to produce waterproof plywood glue. Extensive testing showed it was suitable for construction of exterior grade of plywood. One commercial plant is using soybean meal as an extender for all the phenolic resin used in their mill. These glues may be used with soft and hard woods.

Amylose Triacetate Fibers

The production of fibers from amylose triacetate, previously developed on a laboratory scale, has been extended to the development

of pilot-plant scale dry-spinning equipment and production methods which demonstrate the feasibility of carrying out the operation industrially. Continuous operation has been shown practicable, and spinning speeds up to 400 feet of fiber per minute have been satisfactorily attained. It has also been demonstrated that cellulose acetate fibers can be spun in the same equipment and under the same conditions as amylose triacetate fibers. Fibers of amylose have been shown to be far more extensible than cellulose triacetate fibers, in many cases the former possessing extensibility up to 100 percent. Our work indicates, furthermore, that still greater drafting of the developing fiber is possible and that considerably higher tensile strengths will be possible, thus increasing the potentialities of amylose triacetate fibers.

SYNTHETIC ORGANIC CHEMICALS AND INTERMEDIATES

Formaldehyde Derivatives of Dextrose

A new polysaccharide of high molecular weight has been chemically synthesized from 2,4-3,5-dimethylene gluconic acid. The new polysaccharide is quite insoluble in all ordinary organic solvents. Its limited solubility in a few solvents such as water, formamide and formic acid is accompanied by swelling and a delayed pasting or gelatinization. With alkalis the polymer is readily saponified to give dimethylene gluconic acid. The synthesis, in vitro, of this new polysaccharide, comparable in molecular size to starch and cellulose, is of interest theoretically and practically. A new reaction seems to have been discovered which will be of importance in the field of carbohydrate chemistry. This new polymer shows promise in the fields of plastics, coatings, films, and fibers.

A New Anhydroglucose

D-Glucosan, a byproduct in the destructive distillation of starch, is the first compound definitely shown to have a glycol group resistant to oxidation by periodic acid under conditions used for determining such groups. For hexosans of this type, at least, resistance to periodate oxidation can no longer be considered conclusive evidence of the absence of glycol groups.

NEW AND IMPROVED FEED OR FOOD PRODUCTS

Undevitalized Wheat Gluten

From a survey made by this Laboratory of the potential uses and markets for wheat gluten, it was found that a large potential market exists for undevitalized wheat gluten if it can be produced at a price of 15 cents, or less, a pound.

The present industrial method of drying undevitalized wheat gluten is an expensive batch process requiring considerable hand labor. Since a great potential demand for the product exists, methods of drying atmospherically have been investigated. Experimental runs conducted on a semi-pilot plant scale, and using equipment designed and built in this Laboratory, showed the most successful drying operation to consist of making a film of the wet gluten, drying the film, and then grinding the dried product in a hammer mill to pass through an 80-mesh screen. The product may be made into a dough having a strength equal to or greater than that of commercial gum gluten.

Fermentation Residues as Animal Feeds

The synthesis of vitamins by Penicillium chrysogenum during the production of penicillin by submerged fermentation has fostered considerable interest as to the nutritive value of the residue, since its recovery as a feed would ease a serious waste disposal problem. P. chrysogenum was found to elaborate significant amounts of several B-complex vitamins. The dried residue is being tested by the Bureau of Animal Industry for its value as an adjunct in poultry rations.

Microbiological Synthesis

As a means of increasing the nutritive value of byproducts, resulting from the production of power alcohol, synthesis of vitamins by various microorganisms cultured in thin solubles has been determined. Among the Aspergilli studied, strains of A. wentii were found superior to other species in the production of riboflavin and pantothenic acid. Two- and five-fold increases in these vitamins were observed. The study was later extended to the genera Penicillium and Rhizopus. Among the former, all of which synthesize only small amounts of riboflavin, were some strains capable of marked synthesis of other vitamins. Three-fold increase in pantothenic acid, two- to ten-fold increase in pyridoxin, and two- to five-fold increase in biotin over the concentrations of these vitamins in the original solubles were obtained. Except for riboflavin, some Rhizopus cultures are even more capable of vitamin synthesis. Ten-fold increases of biotin and pantothenic acid and two-fold of pyridoxin and nicotinic acid over the original stillage levels were not uncommon. The finding that many mold species synthesize substantial amounts of B-complex vitamins may allow the development of byproduct feeds of substantially increased value.

A process for the production of riboflavin by Ashbya gossypii has been developed which improves upon that employing Candida flareri.

Higher riboflavin yields are obtained and the iron content of the medium need not be carefully adjusted. Yields of riboflavin of 300 to 500 $\mu\text{g.}$ per ml. which provide cell-free filtrates assaying 50 mg. of vitamin B₂ per gram of dry substance are obtainable

Vitamin Content of Cereal Grains

Since the vitamin content of distillery byproducts is largely derived from the raw material, a survey of the vitamin content of unfermented cereals has been made. Sorghum varieties showed the greatest variation in vitamin content, particularly in nicotinic and pantothenic acids and biotin. In B-complex vitamins the sorghums showed the greatest potency, followed by other grains in descending order as listed: barley, rice, corn. The survey of potencies of cereal grains with respect to B vitamins will allow selection of grains for alcohol production so as to obtain byproducts of improved quality.

EXTRACTION, RECOVERY, AND PROPERTIES OF SPECIFIC CARBOHYDRATES, OILS, AND PROTEINS

Wet-milling of Damaged Corn

Considerable quantities of corn were harvested and stored during the season of 1944-45 with a moisture content of 20 to 40 percent. Much of this corn molded and heated in storage. Wet-millers encountered difficulty in obtaining separation of the starch from the protein, and many appealed to this Laboratory for advice and information. Steeping studies were made with moldy and "mahogany" corn. Processors had reported only that yields of starch were low because of poor separation on the tables and that the starch was of poor quality, containing higher amounts of protein than normal and possessing a bad color and viscosity. Our results confirmed these observations but also showed that inordinately high amounts of starch and protein were retained in the fiber fractions. Our investigations show that damaged corn requires a shorter steeping period than sound corn to reach the same moisture content. Also, because of the fragmentation of the starch granules, it would be impossible to obtain high-quality starch from "mahogany" corn by any method. At present the only practical procedure for handling this material in a commercial plant is to blend it, in small proportions, with sound corn.

Alteration of a Beet Sugar Factory to Produce Glucose Sirup from Flour

When it became apparent during the war that production of corn sirup would be insufficient to supplement completely the dwindling

supply of sugar, investigations were initiated at this Laboratory for working out new processes for the production of starch from wheat flour. As a result the so-called "batter process" was developed, and consideration was given to its installation in altered beet sugar factories to produce glucose sirup from flour. A typical plant with a capacity of 1,000 tons of beets a day was used as a basis. The altered plant would have a daily capacity for processing 144,000 pounds of moisture-free flour with a production of 148,300 pounds of glucose sirup having a gravity of 43° Baume', and 29,700 pounds of devitalized gluten containing 3 percent moisture. One commercial firm used this batter process on a large scale in a beet sugar plant.

Industrial Use of Laboratory Process for the Wet-Milling of Whole Wheat

Because of the dwindling supply of corn reaching the cash market during the summer of 1945, operations in the corn wet-milling plants of the country were curtailed drastically. One of the largest starch manufacturing companies, faced with the possibility of closing its plant, asked the Laboratory for advice on wet-milling whole wheat in a corn wet-milling plant. As a result of Laboratory recommendations, the company ground more than a million bushels of wheat for the production of sirup and byproduct feed. Careful study revealed that only minor changes were necessary in plant equipment in order to utilize wheat in a corn wet-milling plant. Experience shows that no changes are required in operating methods. The quality of the sirup is excellent. Although the cost of producing wheat glucose is higher than for corn glucose, under the conditions which existed in the summer of 1945 the wet-milling of whole wheat enabled plants to continue in operation, producing sweetening agents and livestock feed when both were badly needed.

STRUCTURE AND OTHER BASIC INVESTIGATIONS

Isomaltose

Work on isomaltose (6- α -glucosido-glucose), the presumed anomalous unit in starch, is progressing along the following lines: (a) synthesis by isomerization of gentiobiose, (b) isolation from enzymic hydrolysates of amylopectin, and (c) isolation from the chemical and enzymic breakdown of dextran. Solution of the isomaltose problem would facilitate the determination of the structure of starch.

Retrogradation of Amylose

Further characterization of amylose has been made by determining its rate of retrogradation or the rate at which amylose spontaneously

precipitates from a neutral dispersion. It was found that for a definite type of amylose such as corn amylose the rate of retrogradation increases with the purity of the amylose. The rate of retrogradation of all starches is increased by low temperatures, high solution concentration, poor dispersion conditions, or high amylose content. Of the two components of starch, the amylose fraction has the more pronounced tendency to retrograde and hence may be a factor in the staling of bread. In addition to its fundamental interest, the results of this work have been made available to the Quartermaster's Corps in connection with the Army's bread staling problem.

Structure of Amylopectin

The structure of the amylopectin fraction of starch has not been well established. Furthermore, little has been known concerning differences in the amylopectins from different plant sources. The amylopectin fractions of five varieties of starch were isolated and hydrolyzed with β -amylase to maltose and a residual limit dextrin of approximately one-half the original molecular weight. It was found that in most respects the amylopectins from different plant sources are alike; however, the amylopectins from starches grown above ground differ from those grown below ground in a few properties that can be attributed to the presence of combined phosphorus in the latter class.

Starch Fractionation

The separation of amylose from starch by means of fatty acids has been accomplished on a pilot plant scale. This is the first successful operation of its kind, on a pilot plant scale, yet reported. It is anticipated that it will lead to possible commercial fractionation.

CHEMICALS DERIVED FROM FERMENTATION

Penicillin

The penicillin project has been completed. In the period intervening since 1941, when work started, our research resulted in increasing yields from 2 to 4 units/ml. to 450 units/ml. Production of penicillin has now reached the amazing figure of approximately 700 billion units per month. The quoted price of penicillin dropped from \$20 to 59 cents per 100,000 units. The expected life of penicillin in storage, originally estimated at 3 months, was extended to 1-1/2 years. Although the work on penicillin was considered essentially a war project, its bearing on the utilization of agricultural commodities is shown by the use in media during 1945 of approximately 12 million pounds of corn steep liquor from cornstarch manufacturing

plants, and of 6 million pounds of lactose. The corn steep liquor had a value of \$234,000, and the value of lactose used was \$1,560,000.

Studies on the production of the chloroform-insoluble penicillin, or Penicillin X, in submerged culture have been completed, and the superiority of the strain developed here (Penicillium chrysogenum NRRL 1984.N22, an ultra-violet induced mutation) has been confirmed in large scale laboratory and pilot plant equipment. Industry is now in a position to produce this penicillin in any quantity desired to meet research and clinical needs.

Itaconic Acid

Interesting results have been obtained in studies of the cultural requirements for itaconic acid production. Itaconic acid has been produced from glycerol, glucose, xylose, and sucrose. A surface culture method for producing itaconic acid has been developed which results in yields of about 37 grams of acid from 100 grams of glucose. Submerged culture methods preliminary to development of vat culture techniques have given as much as 33 grams of itaconic acid per 100 grams of glucose. Itaconic acid esters show promise as constituents of many plastic compositions.

Culture Collection

Large numbers of mold cultures continue to be submitted to the culture collection of the Laboratory for correct identification. These have come primarily from laboratories investigating the tropical deterioration of military equipment, and are largely of tropical origin.

Since the termination of the war, contacts have been re-established with the outstanding culture collection, the Centraalbureau voor Schimmelcultures, Baarn, Holland. Exchange of cultures will greatly strengthen our collection of working tools for fermentation studies. All accessions are preserved in desiccated form, and thus a vast reservoir of types from sources that are worldwide is being built up.

SOYBEANS AND OTHER OILSEED CROPS UTILIZATION INVESTIGATIONS

COMPOSITION AS AFFECTED BY RAW MATERIAL PROCESSING AND STORAGE

Changes in Oil Content of Soybeans

A study is in progress of apparent changes that occur in the soybeans during storage. A 10,000-bushel bin was carefully

sampled when it was filled. The beans in this bin, after standing one month, were turned and resampled. No change in oil content was observed. The experiment is continuing in an effort to determine definitely if the oil content decreases during the several months which may elapse between placing in storage and processing.

SURVEYS AND ECONOMIC STUDIES

German Technical Developments

Soon after V-E day an oilseeds expert of the Laboratory was dispatched to Germany on a mission for the Technical Industrial Intelligence Committee. Under sponsorship of the Joint Chiefs of Staff, an investigation was made of the recent technical developments in Germany in the fields of oilseed processing, oil refining, margarine production, and manufacture of synthetic fats. The information obtained, much of which should prove of considerable value to American processors of oilseeds and oils, is now being declassified for release to manufacturers in this country.

FIBERS, FILMS, PROTECTIVE COATINGS, AND ADHESIVES

Catalytic Isomerization of Non-Conjugated Fat Acids and Their Derivatives

In an attempt to find replacements for tung and oiticica oils, this Laboratory initiated research on catalytic methods of shifting the unsaturation in soybean and linseed oils from the non-conjugated to the conjugated form. The research led to the discovery of a nickel-carbon catalyst which readily produces from 25 to 35 percent conjugation in either soybean or linseed oil. Briefly, the process is as follows: Nickel formate solution is evaporated in the presence of specially prepared carbon black and the nickel reduced with hydrogen at 360° C. The catalyst is mixed with the oil and heated at 170° C. for 4 to 6 hours, after which the oil is separated from the catalyst. If the linseed or soybean oil has been alkali-refined, re-uses of the catalyst five or more times is possible. Data from Laboratory investigations on the effects of different methods of operation have been published in Bulletin AIC-101, of this Laboratory.

Evaluation work on these nickel-carbon conjugated oils has shown that they possess certain characteristics similar to tung oil. The polymerization rate of these nickel-carbon conjugated oils is considerably accelerated, and the films of the oils and of paints and varnishes derived from them have superior alkali resistance. In the case of varnish, however, the greater resistance to alkali, as determined by laboratory tests, was not pronounced, and there are other factors inherent in the oils which, apparently, do not make them clearly superior for varnishes when compared with their source oils. However, varnish manufacture is an art, and others may be able to show how the nickel-carbon conjugated oils can be used to produce varnishes of superior quality.

Nickel-carbon conjugated soybean oil paint containing white lead as a pigment was found to dry very rapidly; in fact, faster than linseed oil paint. However, the nickel-carbon conjugated oil paint develops a residual tack which in exterior paints promotes dirt collection. Studies on methods of removing this residual tack indicate that alkaline earth oxides in the paint film remove the greater part of it. Exposure tests indicate that the dirt collection problem with this special soybean oil paint is comparable to that with linseed oil paints. Weathering tests are in progress.

SYNTHETIC ORGANIC CHEMICALS AND INTERMEDIATES

Copolymerization of Fat Acids and Their Derivatives with Other Polymerizable Materials

Soybean oil, containing a high percentage of linoleic acid, represents a potential raw material source for the manufacture of organic chemicals. The discovery at this Laboratory of a nickel-carbon catalyst for conjugation of linoleic acid has led to studies of the condensation of conjugated soybean oil esters with ethyl crotonate. These studies have shown that the ethyl crotonate condenses readily with the conjugated esters from soybean oil to give a mono-unsaturated dibasic acid ester. If non-conjugated esters are used without a catalyst, a mixture of a mono-unsaturated and di-unsaturated dibasic acid esters is obtained. The yield of dibasic acid esters adduct is 76 percent of the total conjugation. Studies on the nickel-carbon isomerization reaction indicated that an equilibrium might exist between conjugated and non-conjugated forms. Work on this phase of the problem is still in progress.

The adduct of ethyl crotonate and conjugated esters will yield simple polyesters and polyamides of 1,000-3,500 molecular weight by the standard methods of polycondensation. Both the simple esters and polyesters of the adduct may find use as plasticizers of vinyl-type polymers.

Studies on Reversion of Soybean Oil

In studies being made on soybean oil by adsorption analysis, it has been confirmed that the yellow color of refined, deodorized soybean oil is caused by carotinoid pigments and to a substance whose absorption spectrum is characterized by a broad continuum which decreases with increasing wave length. When this latter material was further purified it was shown to contain at least two parts; a small amount of porphyrin and a fat acid mixture ranging in color from light to dark brown.

Investigations and studies on flavor indicate that one or more of the numerous minor constituents present in soybean oil may be responsible for flavor instability. Since one part of a constituent in from 100,000 up to 10 million parts may be responsible for off-flavors in oils, improvement in the methods of analyzing for minor constituents are required as research tools.

At present, methods of determining the amount of the phosphatide fraction in crude and refined soybean oils are being investigated. By employing aqueous hydrolysis and water-ether partition the methods of analysis for acid-labile nitrogen and phosphorus have been considerably improved. When further increase in sample size is obtainable, at least a three hundred-fold improvement in the analysis may be attained.

Soybean Flour

Further work on alcoholic extraction of soybeans to produce an improved soybean flour has continued. In a cooperative project with the Federal Soft Wheat Laboratory, it was shown that for bread-baking formulas containing the proper amount of potassium bromate, the blending of 2 to 8 percent petroleum ether or ethanol extracted soy flour with red spring wheat produces an increase in loaf volume. The behavior of the soy flour in the bread is very similar to that of dry milk solids. One sample of petroleum ether extracted flour was superior to ethanol extracted flour in loaf volume, but in taste the ethanol flour was superior.

EXTRACTION, RECOVERY, AND PROPERTIES OF SPECIFIC CARBOHYDRATES, OILS, AND PROTEINS

Extraction of Oilseeds with Alcohol

A new method which may avoid the necessity of dehydrating the alcoholic miscella by distillation is being investigated in large-scale laboratory equipment. The important features of the process are: Extraction of the soybean flakes with 95 percent ethanol under 15 pounds pressure; cooling of the miscella to approximately 0° C. to remove phosphatides, carbohydrates, oil and other constituents; and

recycling of the separated alcoholic miscella for extraction. If flakes containing 3 percent moisture are used in this process, the moisture content of the alcoholic miscella does not increase, and the necessity of dehydration is avoided. The indications are that this process will operate at a lower cost than present methods which use hexane; furthermore, it will be less hazardous to operate and will yield improved products.

Fractionation of Soybean Oil by Liquid-Liquid Extraction

Research is continuing on the development of a commercially feasible process for fractionating soybean oil by liquid-liquid extraction, using the so-called two-solvent system in which the original oil is introduced at approximately the middle of a vertical column through which furfural and naphtha are flowing counter-currently. The highly unsaturated fraction dissolves in the furfural and is withdrawn at the bottom, and the more saturated portion dissolves in the naphtha phase which overflows at the top. Tests have been made in the pilot plant to ascertain the effects of changing such variables as solvent ratio, temperature of solvent and feed, and reflux ratio. The immediate objective is to establish optimum operating conditions for conducting this type of fractionation, so that a reasonably accurate estimate can be made of the cost of operation. It has already been established that the two-solvent method of fractionating soybean oil by liquid-liquid extraction yields much better results than does that employing a single solvent. Additional developmental research must be concluded, however, to permit appraisal of its economic feasibility.

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EASTERN REGIONAL RESEARCH LABORATORY

P. A. Wells, Director

APPLE UTILIZATION INVESTIGATIONS

JUICE PRODUCTS

Sirup and Concentrate

Concentrates for Beverages: During the past year our work on apple juice concentrate has been directed toward the determination of the cause of an unpleasant, broccoli-like off-flavor which develops in most concentrates when they are kept at room temperature. This off-flavor makes the concentrate unsuitable as a base for the preparation of full flavor concentrate. Full flavor concentrate is concentrate to which essence has been added in the proper proportions and which can be reconstituted to "fresh" apple juice by the addition of water. Comparisons of various commercial concentrates, some of which do not develop this off-flavor, have led us to suspect that the variety of apple is an important factor. Therefore, in so far as the very limited apple crop permitted, we obtained varieties of apples covering a range of high and low acidities and tannin contents. These were separately processed in the pilot plant to produce depectinized concentrates under standard conditions and are being systematically evaluated under different storage conditions to determine the effect of apple variety on the keeping qualities of the concentrate. An attempt will be made to obtain a quantitative measure of off-flavor rather than rely on organoleptic tests.

VOLATILE FLAVOR RECOVERY AND APPLICATIONS

Apple Essence

The process reported last year for the preparation of apple essence concentrate has been applied commercially by two American and three Canadian companies, based on information supplied by us. Production was necessarily limited due to the scarcity of apples this past season. Two other manufacturers are installing essence recovery units and eight additional companies have indicated their intention of going into essence manufacture as soon as materials are available. Industrial interest in this development has continued to grow, and from present indications apple essence manufacture will be widely adopted throughout the apple industry.

Orange Juice

In order to determine the feasibility of recovering the volatile flavor constituents of orange juice using the principles developed at the Eastern Regional Research Laboratory on apple juice, the flavor recovery equipment was shipped to the U. S. Citrus Products Station at Winter Haven, Florida, where fresh orange juice was available in quantity. Experiments were conducted there cooperatively with the personnel at that station.

The orange essence, that is, the concentrated volatile flavors in the water phase, which was obtained in these studies is somewhat similar in ethereal character to apple essence; however, at the same relative concentration (referred to fresh juice) it is a much milder flavoring material. Its addition in the normal amount to a drink reconstituted from orange concentrate tends to mask the cooked flavor and other off-flavors which have developed on storage if these flavors are weak. Tests now being conducted by the U. S. Citrus Products Station at Winter Haven will determine if this essence has sufficient value as an orange flavor to recommend its production and utilization.

Essence from Other Fruits and Berries

Through the cooperation of a local preserve manufacturer who installed a pilot plant flavor recovery unit based upon our recommendations, various fruit and berry juices were processed experimentally to determine the conditions for essence recovery, character of the essence and possible use in the preserve industry.

The juices processed were damson plum, black raspberry, blackberry, Montmorency cherry, grape (fresh), grape (pasteurized), quince and apricot. Since the only strawberries and peaches available had been frozen with sugar, the "juice" processed was actually a sugar sirup drained from these fruits. The flavor recovery process in all cases yielded an essence characteristic of the odor of the fresh fruit. Some of them, however, were not particularly pleasant. The outstandingly good ones were the essences of strawberry, peach, fresh grape and blackberry. It was found necessary to vaporize different percentages of the juice to obtain all the volatile flavor. (8% is sufficient with apple juice)

It thus appears from these preliminary experiments that although true fresh fruit essence can be prepared from a wide variety of juices using the process developed for apple juice, this particular method of stripping and reincorporation of the essence is not applicable to the preserve industry. Studies are planned on the recovery of essence from the concentrate obtained in the conventional vacuum concentration of preserves and jams.

VEGETABLE UTILIZATION INVESTIGATIONS

PROCESSING OF VEGETABLE WASTES

Our proposed design of commercial driers for vegetable waste was based upon experience in the pilot plant with a tray drier. In order to obtain more specific information on the performance of a continuous drier on this type of material, the tray drier was converted to a triple belt, through circulation, continuous drier of the same type previously recommended. Performance tests were superior to those using the tray drier and justify our estimates of cost and performance made in Circular AIC-76.

Since the publication of Circular AIC-76 there has been no necessity for further experimental work on the pilot plant drying of vegetable waste. Operations during 1945 were devoted to producing approximately 1,000 pounds of broccoli leaf meal for chick feeding tests, studies on vitamin extraction, and for market development. Approximately 600 pounds of leaf meal were also prepared from lima bean vines for similar uses.

RECOVERY AND PROPERTIES OF VEGETABLE LEAF PROTEINS AND OTHER CONSTITUENTS

Protoplasts

Digestion of vegetable leaves with the organism Clostridium roseum was shown last year to produce leaf protoplasts rich in protein and lipoidal constituents and low in fiber and carbohydrate. It was decided that this method might be valuable for the quantitative recovery of leaf proteins from a number of vegetable wastes. Protoplasts have been obtained so far from four vegetable leaf wastes (beet, broccoli, carrot and lima bean) by digestion with Clostridium roseum at 37° C. for 40 to 44 hours with subsequent screening off of bagasse and settling and centrifuging of the protoplasts. These protoplasts showed protein values varying from 27.9% (carrots) to 61.1% (broccoli), and fat contents of from 9.63% (carrots) to 23.08% (broccoli). By extraction of the fat from the leaf protoplasts, the protein contents have been raised to from 37% (from carrots) to 84% (from broccoli).

RUTIN

Chemical Studies

Studies of the factors influencing the rutin content of buckwheat have shown that the maximum rutin content occurs at approximately the same physiological age, regardless of the rate of growth. This has an important bearing on the question of the optimum date for harvesting the crop. Studies on the extraction of rutin from green buckwheat have shown that rutin may be extracted rapidly and quantitatively by alcohol and that the subsequent recovery and purification present few difficulties.

Clinical Testing

Clinical testing of rutin is being carried out in cooperation with Dr. J. Q. Griffith, Jr. of the University of Pennsylvania. Dr. Griffith has treated a large number of patients with rutin, and the results continue to show that rutin is almost 100% effective in decreasing capillary fragility associated with hypertension. In some instances this is accompanied by a lowering of blood pressure. Dr. Griffith has also been studying the effect of rutin in the prevention of X-ray burns. Preliminary results indicate that in rats buffered with rutin the onset of X-ray burns is delayed, the burns are less severe and heal more rapidly than in non-buffered animals. Another physician has reported favorable results following the use of rutin in certain hemorrhagic conditions and functional bleeding, and Dr. Lorand Johnson of Western Reserve Medical School from extensive clinical studies has indicated that rutin is effective in stopping the progress of diabetic retinitis.

Rutin has been furnished by the Laboratory to more than 100 physicians for clinical testing, and to several large drug firms that planned to distribute it to cooperating physicians for clinical study. Rutin also has been furnished to Dr. Floyd DeEds of this Bureau for pharmacological study with emphasis on the development of a method for its bioassay.

Commercial Production of Rutin

Appreciable quantities of rutin have been prepared in the Laboratory pilot plant to make supplies available for continued clinical testing until commercial production gets under way. Chemical engineering studies are being made in the course of rutin production to determine the most feasible methods for commercial use. This includes the possible use of hot water as an extractant for rutin, which would eliminate subsequent treatment with benzene. Because of the health and fire hazards involved in the use of benzene, elimination of its use for commercial production would be highly desirable.

During the summer of 1945, four pharmaceutical companies began commercial production of rutin based on our recommendation and advice. A number of other companies either are definitely planning or are giving serious consideration to going into production in 1946.

Because of the importance of making larger quantities of rutin available to meet the increasing demands from physicians for rutin for treatment of patients, and for further clinical evaluations, every assistance is being given to interested pharmaceutical companies in getting into production. Where relatively small production is involved it is possible for manufacturers to either grow or contract for the growing of small acreages of buckwheat near the extracting plant, and the green extraction of the plant material can be carried out. Where larger production of rutin is involved, it may be advantageous for the manufacturer to be able to contract for dry buckwheat meal with a specified rutin content. It is hoped that some such arrangement can be effectuated during the coming season.

TOBACCO UTILIZATION INVESTIGATIONS

PREPARATION AND EVALUATION OF NICOTINE INSECTICIDES AND FUNGICIDES

Metal Nicotine and Nicotinium Salts

In connection with studies on the development of new nicotine compounds for use as insecticides and fungicides, a considerable number of metal nicotine salts and nicotinium salts have been subjected to laboratory and field tests for determination of their insecticidal or fungicidal efficacy. Testing was carried out in cooperation with the Bureau of Entomology and Plant Quarantine and various State Experiment Stations.

One of the compounds tested, zinc nicotine thiocyanate, in field tests against the cotton aphid was found to be approximately as effective as the nicotine mixtures and compounds used commercially. On the basis of these preliminary results, the Bureau of Entomology and Plant

Quarantine has requested that a sufficient quantity of this compound be supplied for larger scale field tests next season. Recently completed orchard tests with cuprous nicotine thiocyanate against the codling moth have shown good insect control and negligible foliage injury. While some injury to fruit was noted, it is possible this effect was magnified by the unusually wet season. Tests at the Pennsylvania Station have shown that cupric dinicotinammino fumarate approximates nicotine sulfate in toxicity to the black bean aphid. It was less effective than DDT against the pea aphid in tests at the Wisconsin Station. Laboratory nicotine compound 44 was used as a dust on poultry lice, and the preliminary tests indicate that it is more ^{toxic} than nicotine sulfate and is easier to apply.

In connection with the preparation of nicotinium salts for laboratory insecticide and fungicide tests, particular attention was directed to the study of methyl nicotinium salts and higher N-alkyl nicotinium iodides in view of the previously reported toxicity of methyl nicotinium iodide and dimethyl nicotinium diiodide to the melonworm, southern armyworm, and oriental fruit moth. Recent tests have indicated that the toxicity of the latter two nicotinium salts is attributable, at least in part, to the iodide anion since comparable toxicity to the oriental fruit moth has been shown by the higher N-alkyl iodides. Furthermore, the N-alkyl nicotinium iodides having large N-alkyl groups appear to be less phytotoxic than the homologous compounds with small alkyl groups. The compounds prepared in the course of this study were also submitted to the Rhode Island Experiment Station for fungicidal evaluation.

Laboratory tests by entomologists at the Connecticut Experiment Station have indicated that cetyl nicotinium bromide, and cetyl nicotinium thiocyanate are toxic to red spider eggs. Of the nicotinium salts tested in a preliminary survey, benzyl nicotinium oleate was most effective against the nasturtium aphid while p-nitrobenzyl nicotinium thiocyanate was most toxic to the wireworm. Several nicotinium salts have given favorable results in control of apple scab and the large brown patch fungus in turf, although the duration of protectant action under field conditions was limited, presumably because of the high water solubility of the compounds.

Nicotine-DDT

The phenomenon of synergism between nicotine and DDT which was indicated in preliminary tests was investigated more extensively, and while no conclusive evidence of synergistic action of the mixture was obtained, mixtures containing nicotine and DDT continue to be of definite interest in the field because of the expanded toxicity pattern of the combined toxicants. Use of the mixture results in a lowering of the concentration of DDT on application, which is desirable since smaller amounts of DDT residue remain on the leaves and fruit.

Nicotine-Dusts

Of 38 carriers studied as dust diluents for nicotine sulfate 12 were superior to the pyrophyllite standard against the melonworm and southern armyworm. One carrier, a special type of fuller's earth, was unique not only in its effectiveness as a carrier for nicotine sulfate, but particularly in its ability to adsorb as much as 35% of an animal, vegetable, or mineral oil and still remain dustable. This high oil tolerance should be advantageous in the formulation of other oil-soluble insecticides, such as DDT. As a result of our studies this particular brand of fuller's earth, which was formerly used only as a catalyst carrier in hydrocarbon reactions, is now being sold in carload quantities for insecticidal use.

NICOTINIC ACID AND ITS DERIVATIVES

Water Insoluble Forms of Nicotinic Acid

In connection with studies directed to development of an anti-pellagra vitamin composition suitable for the fortification of food products which are washed prior to cooking, efforts have been concentrated on the preparation of insoluble esters and amides of nicotinic acid. Previous studies had shown that while some of the N-alkyl esters and amides of nicotinic acid were sufficiently insoluble to be satisfactory for the intended purpose, biological tests on dogs indicated only fair activity for the more insoluble compounds as compared with nicotinic acid itself. This would indicate incomplete digestion and assimilation of the compounds.

PYROLYSIS OF NICOTINE

Laboratory studies on the preparation of various compounds obtained through pyrolysis of nicotine have been carried out with a view to obtaining substances of possible use as pharmaceuticals or for other applications. A number of these compounds, including nicotyrine, nornicotyrine, N-methyl myosmine, d,l-nornicotine, and 3-(4 aminobutyl)-pyridine, have been prepared and submitted to outside laboratories for pharmacological testing.

Of especial interest for possible use as an adjunct in tobacco is the compound myosmine. This substance is a constituent of ordinary tobacco smoke, and presumably contributes to the flavor and aroma of the smoke. It was thought therefore that it might possibly be of practical value as an adjunct in cigar, cigarette, and pipe tobacco to increase flavor and aroma. Results of organoleptic tests carried out at the Laboratory were rather inconclusive as to whether or not treatment of tobacco with myosmine improved the quality of the smoke. In most cases tobacco treated with myosmine could be readily detected on smoking, but there was rather wide divergence of opinion among individuals as to whether or not the smoking qualities had been improved by such treatment. Considerable interest in myosmine for this purpose has been exhibited by the tobacco trade, and samples of tobacco

so treated have been submitted to a number of tobacco companies for more complete evaluation by their testing panels.

RUTIN

Studies to determine the suitability of green tobacco as a source of rutin were continued. Since Pennsylvania seed leaf grown at Lancaster, Pa., and preserved green in alcohol was found to contain no more than traces of rutin, there arose the question whether this was due to climatic, varietal, or soil conditions. An experiment was set up in which Pennsylvania seed leaf, Burley and flue-cured tobaccos were grown at Lancaster and were collected while green, immediately immersed in alcohol and transported to the laboratory. The result was that no rutin was found in spite of the fact that flue-cured tobacco grown in its native belt contains up to 0.4 percent of rutin after curing.

A parallel experiment was set up at the Oxford, North Carolina, Experiment Station, in the flue-cured belt, where Pennsylvania seed leaf was grown and, when ripe, the leaves were flue-cured. The material from this experiment has not yet been received.

MILK PRODUCTS UTILIZATION INVESTIGATIONS

MILK PROTEIN STUDIES

Bristles from Milk Proteins

The method of making bristles from casein has been improved by the development and installation of specialized equipment for the extrusion, manipulation and hardening of the fiber. With this new equipment, a more quantitative conversion of casein into fiber is obtained with less labor. Further improvements are directed toward making the fiber in a completely continuous manner. Bristles are being furnished to interested manufacturers for making experimental brushes, and two companies have been licensed to operate under the application for patent covering the process, which has been filed by the Laboratory. One company is proceeding with the construction of a factory for the commercial production of casein bristle fiber for use in making paint brushes. It is expected this new factory will be in operation by April 1946.

Textile Fiber from Milk Proteins

Progress has been made on a number of phases of the problem of producing an improved textile fiber from milk casein. It has been demonstrated that improved resistance to boiling of formaldehyde-hardened fiber results when a second treatment with formaldehyde at higher temperature is employed. Results with high-temperature formaldehyde hardening indicate such a treatment may evolve into a satisfactory finishing treatment for casein fibers.

The Laboratory spinning machine has been modified so as to produce a twisted continuous filament yarn suitable for knitting. All previous fiber produced on this machine was not twisted and could be evaluated in yarn and fabric forms only after converting the fiber to staple and this in turn to staple fiber yarn. These operations required twenty pounds or more of staple since this is the minimal amount necessary to charge the smallest carding machine available to us. The modification makes possible the investigation and evaluation of casein as a continuous filament. Its characteristics of high gloss and even dyeing should constitute a practical advantage in comparison with acetate rayon, which requires special dyestuffs and often reveals uneven dye application.

In connection with studies on the structure of protein fibers, it has been shown that a very small proportion of disulfide bonds between protein chains may have an important influence on the dimensional stability of a protein fiber. The ease of rupture of these bonds is increased greatly by mechanical stress.

Plastics from Milk Proteins

Certain studies on the modification of casein by the introduction of higher fatty acid radicals with a view to lowering the water absorption capacity of the finished plastic material have been essentially completed. The fundamental reaction, acylation with fatty acid chloride in aqueous alkaline solution, has been applied to casein with a series of acid chlorides ranging from acetyl to stearoyl, and to a number of other industrial proteins with palmitoyl chloride. All of the products in the form of molded plastic discs have been evaluated for their water uptake. Of the various products prepared, palmitoyl zein in particular appears to warrant further investigation. Conditions for the preparation of palmitoyl casein with either minimal water absorption or maximal strength have been established, but it has not been possible to incorporate both desired properties at the optimal level for each in a single preparation.

Small plastic pieces with considerable dimensional stability can be molded from casein modified by the introduction of higher fatty acid radicals. This is true because of the good flow of the acylated products when molded with relatively little water as plasticizer.

CARBOHYDRATE STUDIES

Plastics, Adhesives, Coatings, and Elastomers from Lactic Acid

Studies pertaining to the utilization of the carbohydrate constituent of whey (lactose) are directed towards converting lactic acid, a fermentation product of lactose, to acrylic acid derivatives, which appear to have wide application as plastics, adhesives, coatings, and elastomers. Of the numerous new products that have been developed as a result of these investigations, some of the acrylic rubbers appear to have considerable promise for industrial development.

Pilot plant studies on the emulsion polymerization of acrylic esters have developed important practical information that will be helpful in the industrial development of acrylic resins and elastomers. It has been found possible, for example, to increase the latex concentration of the monomer used in the charge to as high as 55%. In industrial production this will nearly double the polymer output of a given reaction-kettle over that using 30% latex. It will also enable the marketing of a much richer latex. From the results so far obtained in the continuous polymerization studies, it appears that this operation can be used successfully to produce acrylic resins and elastomers. Advantages of continuous polymerization include reduced hazards and high throughput.

The study of acrylic elastomers has been facilitated by the production of Lactoprene EV (95% ethyl acrylate-5% chloroethyl vinyl ether) on a pilot-plant scale by one of the large rubber companies. The compounding and vulcanization of Lactoprene EV has been studied intensively with great profit. Improved/compound-^{compounding} techniques and recipes have been developed, satisfactory mold lubricants have been found, and vulcanizates having unusual resistance to heat aging have been prepared. Samples of 2 to 25 pounds of Lactoprene EV copolymer have been sent to more than ten industrial laboratories for study and evaluation.

Acrylic elastomers are inherently stable to aging even at elevated temperatures. Some of the vulcanizates of Lactoprene EV are apparently much superior in heat aging properties to all known elastomers except the Silicones. Even after being heated in the presence of air at 300° F. for 21 days, some of the vulcanizates retain their rubbery characteristics.

As shown by swelling data, Lactoprene EV vulcanizates are highly resistant to hydrocarbon oils. Comparative tests have indicated that Lactoprene is superior in this respect to natural rubber, Buna S, Neoprene, Perbunan, and Paraplex, but inferior to Thiokol and Hycar OR-15.

In addition to resistance to oils, Lactoprene has been found outstanding in stability at elevated temperatures, flex-life, and resistance to oxidation and cut-growth. On the basis of these special properties, it is anticipated that Lactoprene will find application in various special fields.

ANIMAL FATS AND OILS UTILIZATION INVESTIGATIONS

BASIC COMPOSITION STUDIES

Work was initiated on the fractionation of lard and tallow by systematic crystallization from solvents. The results have permitted reasonably accurate estimates to be made of the trisaturated glycerides, and at least close approximations of the disaturated, monosaturated, and triunsaturated glyceride content of lard and tallow. It was found that lard contains much more monosaturated and triunsaturated, and proportionately less trisaturated and disaturated glycerides than does tallow. Further work is under way

to determine the limitations of the method and its application to other fats such as hydrogenated lard.

The procedure developed for the fractionation of fats should serve as a useful means of obtaining appreciable quantities of certain glyceride fractions, consisting principally of a single type or class, e.g., trisaturated, or disaturated glycerides, for special investigations such as a study of their physical properties in relation to plastic range, shortening and creaming power, and consistency. The fractionation procedure may prove to be useful in following changes in glyceride composition during hydrogenation. The data from these laboratory-scale separations should serve as a background from which processes may be developed for manufacturing industrially important fats for specialized uses.

STABILITY AND STORAGE INVESTIGATIONS

Work is being continued on the synthesis of new compounds and their evaluation as antioxidants for fats. Attempts to improve and shorten the procedure used in synthesizing the series of higher alkyl esters of gallic acid have not yet been entirely successful. Preliminary results on the evaluation of these materials as antioxidants not only for storage lard but also for certain baked products are encouraging.

Further studies of the probable course of atmospheric oxidation of pure fat constituents were carried on. The volatile products of air oxidation of methyl stearate at 100° C. were found to consist of methyl ketones of about 7 carbon atom chain length, low molecular weight fatty acids of undetermined composition, small amounts of peroxidic substances, and appreciable amounts of unidentified hydroxy compounds.

PRODUCTION AND USE OF SURFACE ACTIVE AGENTS

Oil Additives Derived from Fats

Arylstearic acids made by condensing oleic acid with an aromatic compound in the presence of anhydrous $AlCl_3$ have been found to have valuable properties, in the form of simple derivatives, as addition agents to lubricating oils. At the request of the Naval Research Laboratory, we have prepared a large number of arylstearic acids for their tests.

The Naval Research Laboratory has found these compounds to have valuable properties as corrosion inhibitors. Dodecylphenylstearic acid is particularly good against salt water, comparing very well with "mahogany acids", the sulfonated by-product of lube oil refining. Xenylstearic acid also increases load carrying capacity. The usefulness of these compounds has been verified in large-scale tests.

Some improvements have been worked out in the method of preparing these compounds, resulting chiefly in reduction of the time required for their synthesis. The program under way at present includes work

on further improvements in the synthesis, the preparation of new arylstearic acids, and the preparation of compounds related to the arylstearic acids.

Improved Emulsifiers for Use in the Manufacture of GR-S Synthetic Rubber

A sensitive and accurate spectrophotometric method was developed in this Laboratory for determining the polyunsaturated fatty acid constituents in fats and soaps. By means of this method, and in cooperation with the Rubber Reserve Company and its collaborators, it was shown that the small amounts of polyunsaturated constituents of the soaps were responsible for variable retardation of the polymerization, and that mild selective hydrogenation of the fat was a remedy for the difficulty. New specifications for suitable soaps were accordingly adopted by the industry, requiring hydrogenation of stocks and rigid control of the polyunsaturated fatty acid constituents by spectrophotometric analysis. The new spectrophotometric method has been incorporated in the Rubber Reserve Company's Laboratory Manual, and will be used by the soap producers and by the rubber manufacturers as a means of testing the soaps. This method has been found to be superior to any chemical test or combination of chemical tests for this purpose.

Development of this spectrophotometric method of analysis and its application to soaps used as emulsifiers in the polymerization of GR-S synthetic rubber were important to the Government's synthetic rubber program. As a result of this collaborative study resulting in the adoption of soaps from hydrogenated tallows and greases as emulsifiers for use in the manufacture of GR-S synthetic rubber, the following advances or improvements have been made: (1) 100 million pounds annually of low grade tallows and greases will be utilized; (2) it is no longer necessary to use edible grades of tallow for the rubber program; (3) a 5 percent increase in production of GR-S rubber, using present plant facilities, was effected; (4) variable polymerization rates in the manufacture of GR-S have been eliminated, permitting a definite production schedule for the first time; (5) the sensitive spectrophotometric method of analysis developed has been found applicable to many other fat and soap problems, since the presence of even small amounts of polyunsaturated fatty acids may be of significance in other industrial, research, and food uses of animal fats. It has been reported to us that one large soap manufacturing company is already finding the method useful in preventing loss from oxidative deterioration in soaps.

PROCESSING STUDIES

Investigation of hydrogenation as a means of improving the physical properties and of increasing the stability of lard was undertaken this past year. A series of samples representing successive stages of commercial hydrogenation of lard were subjected to comprehensive examination as to fatty acid composition,

stability, and physical properties. Attempts are being made to determine also the changes in glyceride composition occurring during the hydrogenation, by means of a crystallization procedure developed in this laboratory.

The complete characterization of the hydrogenation of lard to stearins under selective conditions as employed commercially should be of considerable value to industry by providing a better understanding of the chemical and physical changes that occur during the process.

PLASTICS, PLASTICIZERS, COATINGS AND OTHER INDUSTRIAL CHEMICALS

Dihydroxystearic Acid

Continued studies on the oxidation of oleic acid have led to the discovery of a process for preparing dihydroxystearic acid, which holds considerable commercial promise. We have found that by the use of a solvent such as formic acid or acetic acid catalyzed with H_2SO_4 , available oxygen in H_2O_2 can be used almost quantitatively in oxidizing oleic acid to the corresponding formoxy- or acetoxy-hydroxy acid. Either of these can then be readily hydrolyzed to dihydroxystearic acid.

Dihydroxystearic acid is a compound which offers considerable promise of use in the chemical industry but has hitherto not been available at a reasonable price. A number of chemical companies have already expressed their interest in this synthesis and at least one company has made a pilot plant run with good results.

POTATO UTILIZATION INVESTIGATIONS

STARCH ESTERS AND ETHERS

Preparation of Allyl Starch and Other Ethers

Further improvements in the preparation of allyl starch were made. Experiments with allyl bromide at atmospheric pressure indicated that six times the amount of starch originally used could be allylated with the same amount of etherifying agent. Thus only a slight excess of allyl bromide is required, which is desirable because of cost considerations and the fact that with a large excess of allyl bromide more of it is hydrolyzed during the reaction, giving rise to difficult purification problems. When these experiments were repeated using the cheaper but more volatile allyl chloride, it was found that five times the amount of starch (as compared with the originally published method) can be etherified with the same amount of allyl chloride. These improvements will simplify the process and lower the cost of producing allyl starch.

Evaluation of Allyl Starch

Allyl starch is a high polymeric lacquer material which is capable of drying, free from tack, by evaporation of solvent. In addition, it hardens by oxidation to form a coating which is highly resistant to attack by organic solvents. This exhibition of maximum solvent resistance is achieved mainly by baking the coating at temperatures from 100 to 200° C. for various periods of time. Similarly, reasonable solvent resistance may be acquired merely by air drying for approximately one week at room temperature, provided that oxidation catalysts in the form of paint driers are employed.

Data so far obtained suggest that unmodified allyl starch as a coating material is limited in its field of application to a degree comparable with that of unmodified heat reactive phenolic resins. However, the utility of allyl starch as a lacquer can be broadened considerably to include air-drying applications by the addition of modifying ingredients to improve flexibility and adhesion of the coating.

Development of an Air-Drying Finish

Heat-reactive protective coating materials are restricted in their usefulness essentially to an industry where adequate curing equipment is available. Another larger field of use is by the householder, the independent painter, and the furniture manufacturer, for air-drying coating materials for application to wood surfaces. Here the use of drying temperatures in excess of 60° C. are not recommended because of the danger of upsetting the dimensional stability of the wood, or possibly, in the case of furniture, weakening glue lines of the article. Since allyl starch is capable of developing useful protective properties at room temperature, its consideration as a possible air-drying coating material is amply justified.

The following formulation has been found to be suitable for an interior wood finish: Allyl starch (dry), 100; a non-drying alkyd type resin, 30; xylene, 370; Butyl Cellosolve, 37. Cobalt drier (.075% Co on basis of non-volatile content) is normally added to accelerate hardening. This formulation has been used on wood furniture with promising results. The working, drying, and hardening properties approximate those of the best commercial furniture finishes.

It has been noted that this lacquer does not bond well to old varnish, and for the best results all traces of varnish should be removed from a piece to be refinished with allyl starch.

Industrial Interest in Allyl Starch

Since July 1, 1944, over 350 letters have been received requesting information on allyl starch and other allyl carbohydrates. Eighty-five samples have been sent to various individuals and industrial concerns. Over 100 representatives from industry have

visited this Laboratory to confer with staff members regarding the preparation and evaluation of allyl starch.

CHEMICAL AND PHYSICAL PROPERTIES OF POTATO STARCH

Physical research planned to discover and exploit unique qualities of white potato starch is divided naturally into two categories. One is concerned primarily with the starch granules, the other with the molecular components of the granules. Research in both fields is being pursued.

Potato Starch Gels

Formation of starch gels at low concentration is a granule property. Potato starch gels are elastic and moderately clear, but their strength is easily impaired or destroyed because the swollen granules are fragile. The effectiveness of hydrogen bonding agents in modifying potato starch gels is being measured. It is thought that hydrogen bridges between the added agent and the granules may produce a usefully strong gel structure with less swelling and consequent weakening of the granules. Besides conferring increased strength and stability, the hydrogen bonding agent would be expected to increase clarity by diminishing the refractive index difference between the granules and the continuous surrounding medium. Our experiments to date with the typical hydrogen bonding agent, sucrose, show that the expected improvement in strength and optical character is actually produced. It has not yet been possible by this technique to make gels as clear as gelatin or pectin gels. Development of cloudiness in hydrogen bonded potato starch gels after one or two days standing has not yet been overcome. This deterioration is attributable to the slow precipitation of a lightly solvated phase within the granules.

It has been found that potato starch gels have their maximum strength at pH near 6.2, the natural pH of the starch. Serious gel weakening occurs when the pH differs by one or more units from this value.

TECHNOLOGY OF POTATO STARCH

Storage and Quality Studies

Various practices in the manufacture of potato starch have been studied, and samples of finished starch were collected from all potato starch plants in Aroostook County, Maine in order to study the relationship between the different methods of starch manufacture and the properties of the finished product.

Work undertaken in cooperation with the Maine Agricultural Experiment Station has in view the study on storage of potatoes under controlled conditions of temperature, and will deal primarily with the influence of storage conditions on the starch content of potatoes and the yield of starch in the factory. Quantities of Green Mountain and

Katahdin potatoes, the two leading varieties grown in Maine, were placed in storage at temperatures in the range 34° to 60° F. at the Aroostook Farm of the Maine Agricultural Experiment Station in Presque Isle early in October. Samples are being taken periodically for analysis and starch recovery tests.

TANNING MATERIALS, HIDES, SKINS AND LEATHER INVESTIGATIONS

DEVELOPMENT OF NEW AND POTENTIAL TANNING MATERIALS

Canaigre

Field Studies: In cooperation with the Bureau of Plant Industry, Soils, and Agricultural Engineering, experimental plantings of canaigre are underway at Lubbock, and Winterhaven, Texas, State College, New Mexico and Sacaton, Arizona. These plantings included progeny studies of selected strains, fertilizer requirement tests, propagation by seed, effect of growth periods of one to three years prior to harvest, water requirement tests, and root development studies. The field tests have indicated several root strains of superior quality as regards yield, tannin content and purity. No outstanding improvement has as yet been evident as a result of fertilizer applications. Propagation of canaigre from seed and harvesting after two years' growth have given average calculated yields of more than ten tons of roots per acre per year. The average yearly yields and the tannin contents obtained by this procedure were higher than those produced after one year's growth from seed. However, the extractives were low in purity and not well suited for production of high quality tanning extracts. Canaigre roots produced from native Arizona strains have, in general, been found to be higher in tannin and show higher purities than roots produced from New Mexico stock. Samples of wild roots collected in the vicinities of Tucson and Safford have shown, respectively, 31.6 percent tannin with a purity of 64.2, and 31.5 percent tannin with a purity of 63.8.

Sumac

Comparison of Domestic and Sicilian Sumac: Comparison of the tanning properties of Sicilian and Domestic sumac was made on 165 skivers obtained during a commercial tanning test in which Sicilian and three native species of sumac were used for tanning sheepskin skivers. The final evaluation was made by two men experienced in grading sheepskin skivers. In grading, skin defects were ignored and an effort was made to evaluate the leathers on a basis of the tanning agent only. The results indicate that Rhus copallina - a domestic sumac - produced the best leather and Sicilian sumac next best. They were not conclusive as regards the relative value of Rhus glabra and Rhus typhina.

Scrub Oak

Work is being continued on the development of scrub oak bark as a source of tannin. Scrub oak logs are too small to justify hand-peeling and it is proposed to chip the logs and branches, then make

a mechanical separation of bark from wood. Our Florida cooperators - the Engineering and Industrial Experiment Station of the University of Florida - have made available and are using a portable hog for cutting the logs and branches. When an acceptable means of bark preparation and separation has been established, the work on the collection and preparation of 200 tons of bark for use in extract preparation will be begun.

CAUSES AND PREVENTION OF LEATHER DETERIORATION

Mold Resistant Treatments for Leather

Although the compound covered by Army Tentative Specification AXS-1416 has proved quite effective in protecting leather equipment against molds in field tests and has been recommended for use on ordnance equipment, there are some undesirable features in connection with its use. These include the requirement of a controlled elevated temperature during treatment, loss of solvent by evaporation, the tendency of the leather to "bloom" when exposed under highly humid conditions and an occasional failure of the treatment to give entirely complete protection against molds. In view of these facts, officials of the Army Ordnance Department have thought it desirable to study modifications of this treatment and also the possible use of other fungicides.

To study modifications of the compound covered by Specification AXS-1416, and the use of paranitrophenol and dinitro ortho cresol, nine different treating mixtures were prepared and tested. All of these on a basis of laboratory tests are free from the objectional features noted in compound AXS-1416. If these treatments prove to be effective in field tests and show no toxic effects they will deserve consideration for official adoption.

DEVELOPMENT OF COMBINATION VEGETABLE-ALUM AND OTHER TANNAGES

Alum Retannage

The War Department has set up a comprehensive program of research to secure fundamental information on leather and to aid in attaining military leathers of the highest possible serviceability. This Laboratory is continuing its research on alum retanning of vegetable-tanned leather and is at the same time cooperating with the War Department in its leather research program insofar as it is related to the development of better insole leathers by means of alum retannage.

Preliminary small-scale tanning tests have been made in which the use of the customary alum retanning liquor has been compared with that of liquors modified by the addition of a "masking" agent such as acetate and tartrate salts. There was no change in area of the leathers tanned in the "masked" liquors after boiling one minute in water, but the regularly tanned leather under the same test shrank to about one-half its original area. These results are promising since they point to the possibility of producing a leather for use as insoles that will be area stable and resistant to moist heat.

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SOUTHERN REGIONAL RESEARCH LABORATORY

Walter H. Scott, Director

COTTON UTILIZATION INVESTIGATIONS

COTTON LINT

Structure and Basic Properties of Cotton Fiber

In mid 1945 a project was undertaken in cooperation with the Quartermaster Corps on the development of improved water resistant fabrics for military service, with emphasis by the Southern Laboratory on evaluating, improving and utilizing the swellability of cotton fibers under the influence of moisture. Since the end of hostilities, the application of principles and data obtained will be of even greater interest for fabrics for civilian use.

The swelling of cotton fibers has been studied by measuring ribbon width and thickness, and cross-sectional areas of individual fibers, dry and wet. This property has also been estimated by a special centrifuge technique. The amount of water retained after centrifuging may possibly be used as an index of the amount of swelling.

Comparisons are being made in order to determine the relative swelling capacities of different cottons with particular references to the dependence of this property upon variety, fineness, maturity and other fiber characteristics; also to determine the effects of the purification of cotton upon swelling behavior.

Another technique used in the research to design water resistant fabrics is an "orifice test" on yarns to indicate their relative swelling capacities and suitability for such fabrics. Yarns having a high percentage of thin-walled fibers showed evidence of a greater swelling capacity and are therefore believed to be more suitable for fabrics of this type.

Research is continuing on yarns made from cottons of different properties as represented by differences in wall thickness, degree of fineness, and shape of cross section, to determine the effects of these variations on the relative value of the cottons for swelling to fill known amounts of air space in order to reduce seepage of water. This study includes an examination of yarns of different plies and twists to determine the effects of yarn construction on "orifice" test results, and, in turn, help predict the effect of yarns of different constructions on water resistance, when woven into fabrics. In general yarns of the lower twists are preferred.

Preliminary investigations of the fundamental aspect of the mechanism of cotton swelling by water, using the calorimetric "heat of fusion" method of determining the non-freezing (bound) water and the freezing (free) water in raw cotton fiber, has shown some differences in the ratios, depending on the maturity of the fiber. Similar experiments conducted on rayon have shown a higher percentage of non-freezing water and a lower percentage of freezing water, than in raw

cotton fiber.

A working bibliography has been prepared consisting of 750 abstracts covering the literature for the past twenty years on the swelling of cotton and related subjects - in particular, the relationships between cellulose and water with reference to cotton. With the aid of the Department Library, copies of this bibliography may be made available by the end of 1946.

Four different raw cottons have been examined for organic acids. In the samples examined, the total organic acids varied from 0.77 to 0.91, l-malic acid from 0.32 to 0.57, citric acid from 0.05 to 0.10, oxalic acid from 0.002 to 0.005, and unidentified acids from 0.24 to 0.40 percent of the dry weight. Malic and citric acids have been isolated and definitely identified.

Two dyes of different color and different dyeing characteristics have been found which will differentiate between mature and immature fibers. Samples from lots of cotton which did not manufacture or dye properly were submitted by a cotton mill and it was easily shown by this dyeing test that the difficulty was due to a high percentage of immature fibers. The test may also be used on fibers taken from the bolls of growing plants to indicate the degrees of maturity of the fibers, for example - at different points on the seeds.

Cotton Cellulose and its Derivatives

It has been found that cellulose is rapidly degraded by the action of anhydrous acids in low-molecular weight alcohols. The products of this type of degradation are much more stable toward various reagents such as aqueous alkali than are equally degraded preparations obtained by hydrolysis or oxidation. These products should find use where such stability is desirable, as in pretreatments for chemical modification.

Adaptation of Cut Cotton to Chemical Uses

Demonstrational runs on a pilot commercial-size duplex cotton cutter with accessory equipment, in the spring of 1945 led the Office of Production Research and Development of the War Production Board to contract for the construction of a commercial scale double battery machine capable of cutting both lint cotton and first cut linters, to ease an anticipated demand for chemical cellulose. By November 1945, such a machine had been constructed and set up at a Memphis plant, with Southern Laboratory engineers acting as technical advisors. Large scale cutting tests are planned. This large capacity machine is the outcome of work inaugurated in 1942 to reduce lint cotton to short length fibers to insure adequate supplies of cotton cellulose for use in smokeless powder. As a result of favorable tests on this experimental model, a special Congressional appropriation was granted for the construction of the pilot commercial-size duplex cutter on the basis of whose operation the present commercial machine was designed and built.

New and Improved Cotton Processing Machinery

Present methods of slashing used in processing cotton textiles are

fairly adequate from the production standpoint, but the textile industry recognizes that there is considerable room for improvement in the quality of the product, and an experimental slasher with improved fixtures has been designed and a contract let for its construction. Some accessories, equipment, and materials have already been received.

One of the problems encountered in the Laboratory's research on tire cord has been the uniform stretching of cord at high speed. A stretcher based on the principle of constant weight applied to a dancer, and having a double ratchet operating a variable speed mechanism, has been built and is being tested. This new type of stretcher will speed up tire cord research investigations in the Laboratory and may also prove applicable for commercial use.

Protection of Cotton Yarns and Fabrics against Weather and Rotting

To secure effective protection of cotton yarns and fabrics against the action of weather it is necessary to understand as fully as possible the mechanism of cellulose breakdown when samples are exposed under natural conditions, as well as the manner in which this process is influenced by the presence of various chemical treatments or finishes which the cotton may have received. To this end, both plain and chemically finished fabrics have been exposed to weather, the latter group including representative pigment and other protective treatments, and a systematic examination has been made of the degradation occurring in the various samples. Data recorded include strength and cuprammonium fluidity changes, pH, and analytical determination of residual finishes after different periods of exposure. An attempt is also being made to isolate, as far as possible, individual factors in the composite of destructive forces found in weathering and to evaluate each of these in respect to its effect upon cotton cellulose. In this connection, a detailed study of solar radiations is under way to determine the degrading action of different wave lengths and their influence upon specific chemical treatments of cotton fabrics.

Among the various protective treatments so far examined, mineral pigments such as lead chromate and iron tannate have appeared to be the most effective in resisting the destructive action of sunlight on cotton cellulose. Since the color imparted to fabrics by application of such compounds may limit their range of usefulness in certain cases, attention has also been given to the possibility of utilizing for protective purposes, certain materials that do not noticeably color the treated fabric. For example, a form of urea-formaldehyde resin has been observed to retard the degradation of cotton exposed to carbon arc radiations, and cloth treated with this compound is being tested for resistance to natural weather. Exposures are also being made of fabrics treated with zinc dimethyl dithiocarbamate, using urea formaldehyde resin as a binding agent. Glycerol salicylate, a compound that is reported to screen out ultra violet rays, is also being tested as a possible fabric treatment.

An extensive service trial of cotton sand bags, rot-proofed with copper compounds, completed on October 1, 1945, extended through a period of 19 months and required the processing and manufacture of 165

standard size bags. The test supplied valuable practical information about the relative effectiveness of various proofing agencies in protecting cotton fabrics against biological rotting when exposed under severely degrading natural conditions. It also confirmed satisfactorily the dependability of accelerated soil burial as a method of selecting the most highly resistant rot-proofing treatments. A final report on the test was made to the War Department and a summary of its more important features has been prepared for publication.

The excellent rot-resisting properties of partially acetylated cotton yarns, threads, and cloths, repeatedly confirmed at this Laboratory, were announced in press releases. These announcements have aroused great interest, reflected in inquiries about the process and product, in requests for samples, or for the processing of the inquirers' own goods. As a consequence, small lots or samples of partially acetylated yarns, threads and fabrics have been sent out for tests for various uses to different organizations. A number of new uses have been suggested in the inquiries received. Some of these are: boat-lining, seed bed covers, cloth bags for use in water softening systems, bags for covering hams for overseas shipment, electrical insulation in tropical climates. While yarn and thread have been produced readily in moderate quantities, cloths of full widths in fairly large pieces had offered more difficulty, although the process is being adapted to commercial machinery. An original combination of two dyes, of which one colors unacetylated cotton blue, and the other colors acetylated cotton a contrasting yellow, is utilized to test for completeness and uniformity of acetylation, to supplement chemical analyses.

Development of Accelerated Aging Methods for Cotton Textiles

The importance of having dependable accelerated aging tests for chemically treated textiles has long been recognized. To assist in the development of such tests, the American Association of Textile Chemists and Colorists recently organized a special research committee, including representatives from industry, the Army and Navy, the National Bureau of Standards, and the Southern Laboratory, and planned an extensive cooperative research program. From weather exposures made at New Orleans during the past several years, basic data on degradation of cotton fabrics have been accumulated and performance patterns established to serve as a standard of reference in accelerated aging investigations. Since it was found that the breakdown of cotton fabric produced by standard type electric-arc-and-water-spray accelerated weathering units gave no particular correlation with the effects of natural weathering, other means of artificially degrading cotton were sought.

A process in which cotton fabrics were subjected to alternate periods of heating and wetting was found to give good correlation in respect to the resulting strength and fluidity changes with those observed in similar fabrics that had been degraded by exposure to weather under New Orleans climatic conditions. This artificial aging method, however, did not conform closely enough to the effects of natural weather on some of the chemically finished samples examined

to justify the assumption that it would be generally applicable to all types of chemically treated textiles. It appears probable that it can be employed to the best advantage in combination with other cellulose degrading agencies such as carbon arc radiations, which are now being extensively studied.

Development and Evaluation of Testing Equipment and Test Methods

An improvement in the technique for determining the amorphous-crystalline cellulose ratio has been found in the use of certain basic solvents such as ammonium hydroxide, pyridine, or aqueous monoethanolamine (1:1 and 1:2), which dissolves the humic substance formed and deposited on the surface of the unhydrolyzed residue during the process of cellulose hydrolysis. It appears that monoethanolamine diluted 1:3 with water constitutes the most satisfactory solvent. The residues became lighter or almost white after extraction and the extrapolation curves approached the same evaluations that were obtained from computations based on separate experiments where humic substance was formed from glucose.

A method for evaluating and comparing tire cord has been developed which consists in subjecting the cord to a number of longitudinal and cyclic loadings to remove the "dead stretch" and then measuring the work required to rupture the cord. It has long been known that the observed tensile strength of textiles is a function of the rate of application of the test load. In order to determine the magnitude of this effect in the testing of tire cord, 5 widely different types of cord were tested at rates of loading varying from 1 to 5 pounds-per-second. The stretched cord was least sensitive to change in rate-of-loading over this range, while the unstretched cotton cord and the rayon cord were the most sensitive. Studies of the effect of the rate of loading upon the observed tensile strength of tire cord have aided in properly interpreting the results of strength measurements.

For use in investigations on kierboiling and bleaching of cotton fibers, yarns and fabrics, a satisfactory method for the determination of small quantities of the fatty acids or their soaps has been developed.

Flame-proofing Cotton

A trial run of the emulsion flame-proofing treatment developed at the Southern Laboratory was made at a commercial finishing plant at Norwich, Connecticut. 300 yards of cotton drill, 2.5 yards-per-pound, were desized, kierboiled, and the flameproofing chemicals applied. The resulting fabric was found, as expected, to be very satisfactory both in flame and laundry resisting qualities. The increase in the weight of the cloth due to the treatment was approximately 50 percent, which is believed to be much greater than actually necessary for durable flame resistance by this method. Pressure of business in the plant had not permitted sufficient experimentation prior to the run to insure the application of the desired weight increase.

The U. S. Coast Guard Research and Development Section, at whose suggestion this run was made, was notified that the cloth had been treated and was available for trial test.

Special Finishes to Improve Cotton Fabric Qualities

In the chemical finishing pilot plant of the Southern Laboratory, low grade fabrics, such as part-waste osnaburg and 44 x 48 bag sheeting, have been treated by various processes in an attempt to convert these rather unpromising cotton goods, by the use of modern processing methods, into attractive and useful articles. Sample lots of both types have been desized, kierboiled, bleached, mercerized and dyed with fast colors and treated with a durable finishing agent of the modified cellulose type. Results so far secured have indicated a considerable range of promising uses for such goods, including use as garments, draperies, slip covers and the like. These experiments on semi-plant scale of low grade cotton fabrics have indicated interesting commercial possibilities.

Improved Methods of Bleaching Cotton Textiles

A study has been made of 12 regular bleaching processes at 4 commercial plants. Operating conditions were observed; about 70 samples from various stages of processing were collected; and physical and chemical analyses of these are nearing completion. The data acquired will serve as a reference for comparison with newer bleaching methods being developed in industry and for future work in the Southern Laboratory on the improvement of bleachery practice and quality of output.

Economic and Technological Surveys and Appraisals

Cooperative economic research work has been continued with other agencies in a program sponsored by the Marketing Sub-Committee of the House Committee on Agriculture for the purpose of developing factual information needed as a basis for formulating policies and programs directed toward the solution of the cotton problem. Considerable effort was devoted to Project No. V of this program, "Present Status of Synthetic and Other Substitute Products". In this connection an intensive study was made of factors likely to influence the price of one of cotton's principal competitors, rayon, during the next 3 to 5 years. Special attention was given to such factors as raw materials and labor costs, technological progress in the rayon industry, profits involved in rayon manufacturing, and rayon pricing policies.

Many short time surveys were made on subjects such as the following: Value of cotton waste as a by-product of textile manufacture; cotton waste as a filler for plastic molding compounds; wartime improvements in the properties of cotton goods; suitability of certain selections for cotton textile manufacturing; and preparation of lists of manufacturers of such products as cotton twine, cordage, and batting. A study was made, and information furnished the Secretary of Agriculture, for the preparation of a recommendation to the Reconstruction Finance Corporation for the disposition of plants owned

by them which were converted from regular production of cord for rayon tire fabrics. Data was obtained on the cost of producing rayon tire cord yarn in plants in the above category and in plants designed for the production of rayon tire cord.

The Use of Cotton for Tire Cord

Research on cotton tire cord, continued as a principal work of the Southern Laboratory on lint cotton, is now directed wholly to the use of cord in tires for civilian use, in which the requirements are distinctly different than those for military use.

These service tests were made on tires for civilian use during the summer and fall of 1944 by the Government Tire Testing Fleet at San Antonio, Texas, on 6.00-16 passenger car tires, and on 7.00-20 light truck tires run on front and rear wheel positions. Two companies cooperated in the manufacture of these tires, each company producing corresponding sets of tires. Three types of cord were used, a typical commercial type cord; cord manufactured from the Wilds variety cotton; and rayon cord (used in the truck tires only). In the passenger car tire test the commercial cotton cord, as well as the Wilds cotton cord, proved entirely adequate for high speed driving, as no fabric failures occurred in any of the tires. One set of commercial cotton fabric tires and one set of Wilds fabric tires, with one recapping, were driven a total of 68,000 miles at 60 m.p.h. on highways out of San Antonio and the carcasses were still in good enough condition for another recapping. In the light truck tire test, tires in rear wheel positions, made from Wilds cotton cord, gave 3 times the mileage obtained with the tires from the commercial cotton cord. The rayon cord tires gave 16 percent more mileage than the Wilds cotton cord tires, but the results are inconclusive in determining the relative merits of special variety cotton and rayon in this size tire because no fabric failures occurred in either group. Likewise, in the front wheel truck tire tests no fabric failures occurred. Although the nature of the failures in these tests was such that very little information was obtained as to whether rayon is better than special variety cotton for use in tire cord for truck tires, much valuable information was obtained which is proving helpful in further research on the design of improved types of cotton tire cord.

After removal, in November, 1945, of restrictions on the dissemination of details of the above tests, summaries were prepared and published.

Cotton meets its severest competition from rayon in large-size truck and bus tires. With the cooperation of a large tire manufacturer, four groups of 9.00-20 truck-and-bus-tires have been manufactured with the following types of fabrics; (1) commercial-type cotton cord, (2) cord made from Wilds cotton, using a typical commercial process, (3) Wilds cotton cord produced according to the dual-stretching process developed at the Southern Laboratory, and (4) rayon cord. All the cotton cord is of the high-gauge type (large diameter) and principal interest in this test will be the relative performance of the cotton cord tires, since it is not expected that the high gauge cotton cord tires will perform as well as the low-gauge, thin-carcase rayon cord

tires. The rayon cord tires are being included in the test in order to obtain up-to-date technical data on the performance of rayon tires which will be of aid in further research on the development of improved cotton cord tires.

A machine is being constructed with which plies for tires can be manufactured completely at the Southern Regional Laboratory from small quantities of cord, and the plies shipped to tire manufacturers for immediate manufacture into tires. It is anticipated that this machine will cut down the time required for service test cycle by at least 50 percent.

An apparatus and method have been developed at the Southern Laboratory for measuring the dynamic elastic properties of tire cord, which are extremely important in determining tire performance. Significant differences have been observed between various types of cotton, rayon, and nylon cord and this information is being used in designing and evaluating new types of experimental cotton cord. Useful data have also been obtained, through the development of special laboratory tests, on the influence of heat, moisture content, fiber strength, and cord construction on such cord properties as strength, elongation, plastic flow, and flex-fatigue life.

An Improved Cotton Bandage Fabric

Production of moderate quantities of the semi-elastic bandage has been continued to supply the U.S. Naval Hospital in New Orleans, where a total of 25,000 bandage strips has been used. Several surgeons from that hospital and from other hospitals have expressed a real desire for this type of bandage for civilian use. One large bandage manufacturer advised that for the present they have deferred action on plans to produce the bandage commercially on account of the high estimated cost of a pilot plant. On the other hand, it has been reported that another large bandage manufacturer is working on the production of the bandage on a pilot plant scale.

Unlined Cotton Fire Hose

The project on unlined cotton firehose has been active, in the sense that a very large amount of work on the principles involved in making such fabric structures has been directed to army oxfords and similar lighter weight civilian goods rather than to firehose. This work applies to any water resistant fabric which depends wholly or in part for its effectiveness upon self-sealing, the closing of its structure upon wetting through swelling of its constituent fibers to prevent passage of water.

A pilot plant machine has been assembled for applying a supplementary swellable material (Coglin) to yarn in accordance with the principle previously worked out and patented for use in firehose. Yarns thus treated will be woven into fabrics of the army oxford type of different designs.

COTTONSEED

Storage Investigations

Research on improving the storage properties of cottonseed has continued with laboratory investigations on chemical inhibition of biological activity and the development of methods for application of inhibitors on a mill scale. During the past year a laboratory method has been developed to determine rapidly and quantitatively the efficacy of an inhibitor on the heating, respiration, and lipolysis of seeds. The effect of many chemicals has been studied in the laboratory by this method, and ammonia (reported previously) and an aryl alkyl sulfonate, the two which exhibited greatest activity, have been tried in oil mills on a pilot commercial scale. Although results to date have not demonstrated the complete feasibility of commercial application of these two chemicals, the experience gained to date has been of great value in planning and conducting further work with other chemicals which have exhibited much greater and more selective activity in the prevention of seed deterioration during storage.

Preparation and Processing

In the pilot plant the continuous cottonseed and peanut preparation plant was operated to clean, delint, hull, purify, crack, and flake, in two roll mills, several thousand pounds of cottonseed for experimental work.

Pigments

For many years gossypol was the only pigment associated with cottonseed. The Southern Laboratory's research in this field has led to the identification of three other pigments; gossypurpurin, purple in color, and gossyfulvin, orange in color, from raw cottonseed, and gossycaculin, blue in color, from cooked cottonseed.

Microscopic investigation of the distribution of the predominant pigments in cottonseed tissue has shown that they are concentrated in distinct organs of the seed, i.e., in pigmented glands. These pigment glands are mechanically strong, resist the actions of many organic liquids, and have a density less than that of other cottonseed tissue. With this knowledge, a process was devised for the mechanical removal of pigments from cottonseed which consists in floating the largely intact glands on the surface of a mixture of organic liquids having a density intermediate between that of the glands and that of the other seed tissue. A glass fractionation unit of pre-pilot plant scale has been constructed for the removal of pigment glands. Trial runs have been conducted using cottonseed flakes, defatted by solvent extraction with Skellysolve B, and as a liquid for the fractionation process a mixture of tetrachlorethylene and Skellysolve B adjusted to a specific gravity of 1.378 at 27°C. For the first time a sufficient quantity of cottonseed pigments will be available for a determination of the physical, chemical and toxicological properties of these coloring matters, and a study of their functions in relation to seed maturity, seed storage, processing conditions and industrial utilization, and toxicological and nutritional factors.

COTTONSEED OIL

Improvement of Stability

The role of tocopherol as an antioxidant has been further investigated. Using alkali refined and bleached cottonseed oil a series of samples were hydrogenated selectively and nonselectively to produce fats with iodine numbers ranging from 105 to about 30. Analyses were made for saturated and unsaturated acids. For comparative purposes two other series of fats which are free of tocopherol were prepared, one from chicken fat and one from hog fat. Information on the stability of these fats will contribute to a more complete understanding of the effect of natural antioxidants and relative degree of hardness on the keeping quality of processed fats similar to those employed in the manufacture of shortening, margarin, etc.

Laboratory Investigations to Improve Utility and Industrial Application

A contribution has been made to the hydrogenation technology of fats by determining the relative reactivities of different fatty acids toward hydrogen using a recently developed spectral method of analysis for polyethanoid fatty acids.

Research has continued on the synthesis of artificial fats, particularly low-melting fats of limited plastic range, the so-called "hard butter" type. Reactions involved in the preparation of mono- and diglyceride fats with saturated and unsaturated acids have been studied.

Solvent Extraction

A pilot plant investigation was conducted on a lot of prime cottonseed to compare and evaluate the quality of cottonseed oils and meals from hydraulic pressing with those obtained by the solvent extraction of flaked meats, cooked meats and pressed cake. An export model French oil mill unit was used for cooking and pressing operations. Each extraction was performed under identical conditions of temperature and rate. Flavor tests on the refined oils showed that the hydraulic pressed oils were prime; that the solvent extracted oils had a characteristic flavor which may not be objectionable; and that although solvent extracted oil may actually be prime it cannot be so graded, since no known standard has been established for the flavor evaluation of solvent extracted cottonseed oil. Previous work indicates, however, that this characteristic flavor can be removed by deodorization processes presently used for hydraulic pressed oil. Results show that better color and lower refining loss are obtained with the oil from the solvent extraction of uncooked flakes. Cooking prior to extraction has no notable effect on the refining loss but produced a slightly darker refined and bleached oil. The oil solvent extracted from the hydraulic pressed cake had a higher refining loss and was considerably darker than all other oils obtained. The modified refining procedures for oil from solvent extraction of uncooked flakes appear to be equally satisfactory for oil from cooked flakes.

The installation of major and accessory units of a pilot plant for continuous solvent extraction of 200 pounds of meats per hour is nearing completion.

SWEETPOTATO UTILIZATION INVESTIGATIONS

Starch Manufacture and Utilization

The first car of sweetpotato starch from the new factory of the United States Sugar Corporation at Clowiston, Florida, left the plant during December, 1945. An important new milestone was marked in the advance of southern agricultural and industrial enterprise, based on the results of scientific investigation. The new plant, modern and well-equipped in every respect and representing, with associated facilities, an investment of over \$7,000,000, is the outgrowth of over 15 years of research and development work by the Bureau and the Southern Laboratory, on the manufacture of high quality starch from sweetpotatoes. The processes and procedures developed and applied in the Laurel Starch Plant have been translated to large volume operations employing the most recently developed equipment for conduct and control of continuous processing.

After about three months of necessary test runs the factory went on a production basis in January 1946, with an output of about half the maximum of 120 tons of starch a day. Heavy damage to the sweetpotato crop as a consequence of inundation with water during a hurricane in September, 1945, curtailed the supply of material available for this season's processing; but production will continue at least through March.

Major efforts were directed to technical advice and assistance to the new Florida enterprise in setting up operating and control procedures for the manufacturing conditions anticipated and in adjustments of process and equipment to the conditions encountered in actual operation. A number of problems arising from the specific characteristics of the glades-grown sweetpotatoes had to be solved on the ground or referred to this Laboratory for investigation. The experience and data acquired in the Southern Laboratory and Laurel factory, and methods developed for chemical control of processes, and for evaluation of quality of products, have found and are continuing to find most effective application in the new operations. While, as in any new factory, some further adjustments must be made to attain maximum yield and quality of products, the objective of contributing as soon as possible to relief of the current scarcity of starch with an acceptable product has been achieved through co-operative technical effort.

Continued investigations of the progressive reduction in hot paste viscosity during acid modification of sweetpotato starch, and of the effects on starch characteristics of different extraction, purification, and finishing processes in the factory have facilitated such control of starch characteristics in manufacture that products best adapted to specific end uses can be uniformly turned out as required. Cooperative tests in a cotton mill which has been using sweetpotato starch for warp sizing since 1936, have

afforded data on the conditions of composition, temperature, consistency, and pH during preparation and use of the size mixture found most satisfactory for slashing.

With the continued heavy demand for dehydrated sweetpotatoes for military, and later, UNRRA requirements, Sweet Potato Growers, Inc., at Laurel, Mississippi, suspended production and processing of sweetpotatoes for starch in the 1945-6 season and concentrated all efforts on dehydration for food use.

Byproduct Recovery and Utilization

The production of feed yeast from the soluble sugars in sweetpotato starch factory waste water was carried out on sub-pilot plant scale in 50-gallon propagators, using both batch and continuous processes. Yields equivalent to 40-45 percent of the sugars available were consistently obtained, as forecast by the laboratory trials. The continuous process developed, using 2 or 3 propagators in series, affords a practically automatic system which reduces by 2- or 3-fold the tank capacity requirements for a given output and eliminates the need for incremental build-up of culture inoculum necessary for each tank in a batch system. The process can be carried on indefinitely, unless accidental contamination occurs. The air requirements for aeration in this yeast propagation process were found to be much lower than indicated by previous experience, materially reducing the cost of the operation.

Continuous coagulation and concentration of the natural protein material in sweetpotato starch fruit water was effected in improved equipment on a large enough scale to demonstrate its practicability for plant-scale operation. Investigation of the constitution of the protein has been essentially completed, with confirmation of previous findings as to identity and content of specific amino acids. In addition, methionine and cystine have been identified and determined. The protein appears to have good nutritional value from the standpoint of amino acid composition.

The feasibility of propagation of feed yeast on sweetpotato starch fruit water, after heat and acid coagulation of the crude protein but without separation of the latter, and simultaneous recovery of the yeast and protein in a single operation as a protein feed concentrate, has been demonstrated. The process has been carried successfully to pilot plant scale without impairment of the yield of yeast; and it offers promise of materially reducing the processing costs where merely a crude feed protein is required, rather than pure yeast or separated sweetpotato protein.

Based on results obtained with 50-gallon propagators, a complete pilot plant, with all accessories and controls for continuous and practically automatic coagulation of the natural protein in sweetpotato starch fruit water and continuous propagation of *Torula* yeast in three tanks of 500 gallon fermentation capacity each, has been assembled and operated. The plant is designed for production of yeast alone or for combined recovery of yeast and sweetpotato

protein. It is so set up that it can readily be taken down, transported, and set up for experimental operation in a commercial plant. Such trial is contemplated at the new starch factory in Clewiston, Florida, after the starch manufacturing operations are fully established.

Meanwhile, the process and equipment is being applied by other units of the Bureau, in cooperative trial runs for the production of feed yeast on citrus pulp press juice.

Dehydration for Feed, Food, and Industrial Uses

In continued cooperation with Sweet Potato Growers, Inc., at Laurel, Mississippi, equipment was designed for pre-heating sweetpotatoes before lye peeling for dehydration, in order to curb discoloration due to catechol oxidase reaction. The pre-heater is essentially an enlargement of the rotary lye peeler designed in 1943; and with its aid the potatoes are continuously and automatically immersed for about 30 minutes in water held at a constant temperature of about 130° F. before delivery to the peeler. Blackening was practically eliminated. With this improvement in processing and the high quality of the sweetpotatoes available, the peeling and trim losses in the past season were brought to a very low figure and the former bottleneck at the trim table was eliminated.

The plant completed operations on sweetpotatoes for the 1945-6 season at the end of January, with a total production of around 1,350,000 pounds of excellent quality product. An average daily output of about six tons of finished material was sustained and the average raw material: dry product ratio for the entire season was slightly under 6:1. The combined dehydration and raw sweetpotato marketing operations of the Cooperative for the season afforded a profitable outlet to growers in the section for some 156,000 bushels of sweetpotatoes.

Preliminary examination was made of the waste accumulations or effluents of sweetpotato dehydration or canning plants from the viewpoint of byproduct recovery and waste disposal. The solid trim or cull waste or the solid peeling waste where steam peeling is used can advantageously be dried for feed as a number of plants are already doing. Otherwise the waste effluents appear to offer little promise for byproduct recovery and are mainly a disposal problem.

PEANUT UTILIZATION INVESTIGATIONS

PEANUT OIL

Basic Chemical and Physical Properties

A calorimetric examination of the homologous series of the triglycerides from trilaurin to tristearin was completed. The heats of fusion and heats of transition of the liquid and solid forms were determined.

Improvements of Stability

An investigation was carried out on the stabilizing action of proline which was furnished by the Western Laboratory for evaluation of its antioxidant properties. It was suspended in alkali-refined peanut oil prior to deodorization and also in alkali-refined and deodorized peanut oil. The stability (keeping quality) of the oils was more than doubled by the addition of 0.1 percent of proline but its addition imparted a disagreeable odor. This substance appears to act as a synergist and not an antioxidant. Unfortunately, the objectionable odor associated with proline cannot be removed by steam deodorization, which militates against its use in edible oils, although it may have merit for use in non-edible oils.

In studying flavor deterioration in peanut butters and the possible effect of oxygen in initiating flavor deterioration, it was found that butters stored under vacuum at room temperature remained perfectly fresh and palatable during 16 months. Control samples containing a small amount of air became "flat" in taste, lost their pleasant aroma, and developed a certain degree of staleness.

Paralleling the work on cottonseed oil, an investigation has been made between the stability and fatty acid composition in a series of hydrogenated peanut oils. For purposes of comparison, especially with regard to the production of isolinoleic acid during hydrogenation, linseed oil was hydrogenated under selective and non-selective conditions. From the stability test it was observed that the rate of oxidation of isolinoleic is much lower than that of normal linoleic acid and substantially equal to that of normal oleic acid. An unexpectedly high order of stability was encountered in some fractions representing early stages of hydrogenation.

Laboratory Investigations to Improve Utility and Industrial Application

A number of waxlike products were prepared by the reaction of stearic acid with amino alcohols and amino diols. The fatty acid and amino alcohols were reacted in an effort to produce ester-amide linkages in compounds containing 39 to 57 carbon atoms. The purified compounds were hard, glossy, waxlike solids. Samples have been submitted to commercial companies for evaluation.

The properties of fatty acid chlorides, intermediates for the preparation of synthetic glycerides, have been investigated. The relative rates of hydrolysis of the saturated and unsaturated fatty acid chlorides were determined. The rates of hydrolysis of the saturated fatty acid chlorides decrease with increasing chain lengths up to and including C_{12} (lauric acid). With saturated fatty acid chlorides of greater chain length than C_{12} , the rate of hydrolysis becomes progressively greater as the length of the carbon chain increases.

Fractional Solvent Crystallization

In previous years reports were made on the production on a

laboratory scale of the solvent fractionation of cottonseed and peanut oils for increasing their interchangeability with other fats and oils, as for example, olive oil as worsted lubricant. Pilot plant equipment has been procured and installed for low temperature continuous crystallization and fractionation. A series of runs on refined peanut oil have been made in an attempt to establish operating temperatures, through-put rates, etc. A major difficulty has been the pressure build-up in the filters due to the slushy consistency of the filter cake. In an effort to overcome this difficulty, a continuous rotary pre-coat filter unit has been purchased and will be substituted for the batch filters in future runs.

PEANUT MEAL AND PROTEIN

Fiber Production and Processing

Peanut protein fiber research has been continued and an improved machine with greater mechanical flexibility and with provision for a wide variety of chemical treatments of the yarn has been designed and constructed for extruding protein fibers of low denier. With this laboratory-scale machine, the effects of various chemical and physical treatments have been investigated. Fibers produced from peanut protein solution having a pH value near 12.0 have better overall serimetical characteristics than fibers produced from solutions of lower or higher pH. The addition of 1% of either diglycol laurate or oleate to the protein solution before spinning produces an open yarn having a soft hand. Yarns treated with alkali halide solutions have good boil resistance. Evidence has also been obtained that peanut protein yarns loaded to values below the breaking load will creep and that, on relaxation of the load, part of the creep is retained. After rewetting and drying, the yarn will regain its original length without suffering severe damage. It has been demonstrated experimentally that it is feasible to blend peanut protein fiber with natural wool prior to subjecting the mixed yarn to the commercial treatment with sulfuric acid, known as carbonization. Overall results obtained indicate continued improvements in the properties of artificial yarn produced from peanut protein and offer increasing assurance that a commercially valuable fiber can be produced.

Adhesives, Sizings, Coatings, and Plastics

It has been reported previously that peanut protein from solvent extracted peanut meal is highly adaptable for adhesives and glues in such applications as gummed paper tape, book-making, box-binding, etc.

A major drawback in the industrial use of peanut protein has been the color imparted to the finished product by the pigments in the skins. An inexpensive means for removing this color from red skinned peanuts has been devised to permit preparation of peanut protein relatively free of color. This is accomplished by

washing the shelled nuts with 0.5 percent sodium hydroxide solution at room temperature, rinsing and drying before processing for oil and meal. This development eliminates the necessity of using white skinned peanuts to obtain peanut protein of satisfactory color.

Progress has been made in developing information, formulas, and processes for using commercial peanut meal in plywood glues. Experiments were carried out to determine the influence of processing conditions. From this investigation the following specifications have been prepared for meals which give good results when used as plywood glue:

Protein (N x 6.25).....	50% minimum
Percentage of total Meal soluble in 1N NaCl....	70% minimum
Sieve test.....	30% or more through 200 mesh
Oil	6% minimum

Peanut meal glue meeting these specifications was tried on a plant scale by the Mississippi Plywood & Veneer Company. The glue performed well through the process and gave as good or better results as did commercial soybean meal glue which was used to glue the same lots of veneer.

Peanut meal glue is equal to soybean meal glue when tested according to the official birch plywood test and also when tupelo and red gum are used.

In allied investigations additional data has been obtained on the solubility and fractionation of the protein and the behavior of phytin and phytases in the peanut. Viscosity measurements are indicated as a means of control in evaluation of the production, processing, and utilization of protein preparations for specialized purposes. Further investigation has been made of the protein water relationship leading to improved methods for the production, by dewatering peanut protein curds, of peanut protein hydrates reported previously. This process reduces the drying costs and provides a protein of relatively uniform ash content.

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